FILE COVERS 1907 - 12 Apr 2005 VOL 142 ISS 16
FILE LAST UPDATED: 11 Apr 2005 (20050411/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=>
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L1 STRUCTURE UPLOADED

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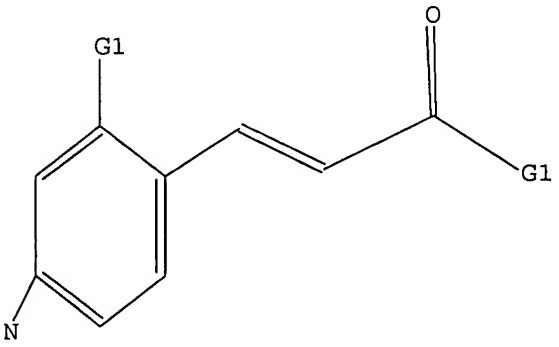
L1 HAS NO ANSWERS
L1 STR



G1 O,NH,S

Structure attributes must be viewed using STN Express query preparation.

=> s l1 full
REGISTRY INITIATED
Substance data SEARCH and crossover from CAS REGISTRY in progress...
Use DISPLAY HITSTR (or FHITSTR) to directly view retrieved structures.

FULL SEARCH INITIATED 14:04:52 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - 1172 TO ITERATE

100.0% PROCESSED 1172 ITERATIONS 742 ANSWERS
SEARCH TIME: 00.00.01

L2 742 SEA SSS FUL L1

L3 103 L2

=> s l3 and py<2001
20649732 PY<2001

L4 86 L3 AND PY<2001

=> d 61-86 ibib abs hitstr

L4 ANSWER 61 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN
ACCESSION NUMBER: 1974:84703 CAPLUS
DOCUMENT NUMBER: 80:84703
TITLE: Yellow coumarin dyes
INVENTOR(S): Sato, Katsunobu
PATENT ASSIGNEE(S): Sumitomo Chemical Co., Ltd.
SOURCE: Jpn. Kokai Tokkyo Koho, 5 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

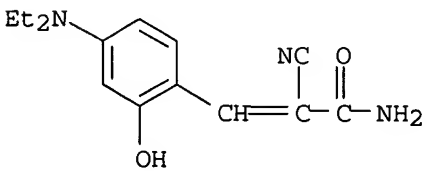
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 48080122	A2	19731026	JP 1972-11985	19720201 <--
JP 51042611	B4	19761117		

PRIORITY APPLN. INFO.: JP 1972-11985 A 19720201

AB Coumarin dyes (I, R1, R2 = H, alkyl, or cycloalkyl, or R1, R2, and N form a heterocyclic group; X = S, NH, or NR3, R3 = alkyl, aryl, or aralkyl; A = benzene or naphthalene ring with or without substituents except CO2H and SO3H) are prepared through condensation reactions. The dyes are useful for dyeing acetate, polyester, or polyamide fibers in fluorescent yellow shades with good fastness. Thus, NCCH2CONH2 was treated with 4,2-(Et2N)(HO)C6H3CHO in MeOH containing piperidine at room temperature to give 4,2-(Et2N)(HO)C6H3CH:C(CN)CONH2 which was treated with o-(H2N)2C6H4 in DMF at 100-10.deg. to give a yellow dye (I, R1 = R2= Et, X = NH, A = benzene ring) [27425-55-4]. Similarly prepared were 2 other I.

IT **42005-48-1P**
RL: IMF (Industrial manufacture); PREP (Preparation)
(preparation of)

RN 42005-48-1 CAPLUS
CN 2-Propenamide, 2-cyano-3-[4-(diethylamino)-2-hydroxyphenyl]- (9CI) (CA INDEX NAME)



L4 ANSWER 62 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN
ACCESSION NUMBER: 1973:527391 CAPLUS
DOCUMENT NUMBER: 79:127391
TITLE: Methine compounds and their coumarin dye derivative
INVENTOR(S): Ikeda, Tsuneo; Sato, Katsunobu; Sugiyama, Hiroshi
PATENT ASSIGNEE(S): Sumitomo Chemical Co., Ltd.
SOURCE: Ger. Offen., 23 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2263822	A1	19730705	DE 1972-2263822	19721228 <--
JP 48071420	A2	19730927	JP 1972-3484	19711228 <--
JP 51007488	B4	19760308		
JP 48103542	A2	19731225	JP 1972-37916	19720414 <--

CH 554366	A	19740930	CH 1972-18857	19721227	<--
BE 793447	A1	19730416	BE 1972-125936	19721228	<--
NL 7217723	A	19730702	NL 1972-17723	19721228	<--
FR 2170618	A5	19730914	FR 1972-46744	19721228	<--
IT 974354	A	19740620	IT 1972-55074	19721228	<--
GB 1404373	A	19750828	GB 1972-59951	19721228	<--
ES 410441	A1	19760501	ES 1972-410441	19721228	<--
CA 1002950	A1	19770104	CA 1972-160070	19721228	<--
US 3914273	A	19751021	US 1972-319816	19721229	<--

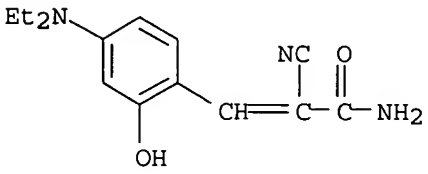
PRIORITY APPLN. INFO.:

JP 1972-3484	A	19711228
JP 1972-37916	A	19720414
JP 1971-3484	A	19711228

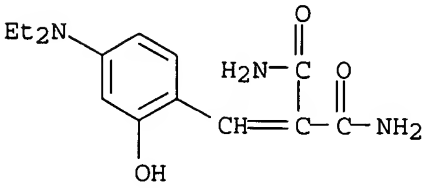
AB The dye (I) [28754-28-1] dyeing polyester, polyamide, and acetate fibers wash-, sublimation-, and lightfast yellow shades was prepared by reaction of 2,4-HO(R2N)C6H3CH:CR1CONH2 (II, R = Et, R1 = CN or CONH2) with isatoic anhydride or with 2-H2NC6H4COX (X = OH or NH2). Four II (R = Me, Et, or MeOCH2CH2; R1 = CN or CONH2), e.g. 2-cyano-3-[4-(diethylamino)-2-hydroxyphenyl]acrylamide **[42005-48-1]** were prepared by reaction of 2,4-HO(R2N)C6H3CHO with R1CH2CONH2.

IT **42005-48-1P 50745-32-9P 50745-33-0P 50745-34-1P**
RL: IMF (Industrial manufacture); PREP (Preparation)
(preparation of)

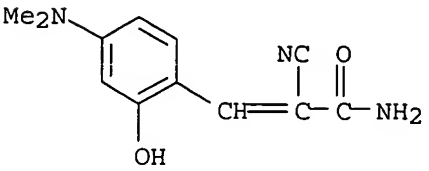
RN 42005-48-1 CAPLUS
CN 2-Propenamide, 2-cyano-3-[4-(diethylamino)-2-hydroxyphenyl]- (9CI) (CA INDEX NAME)



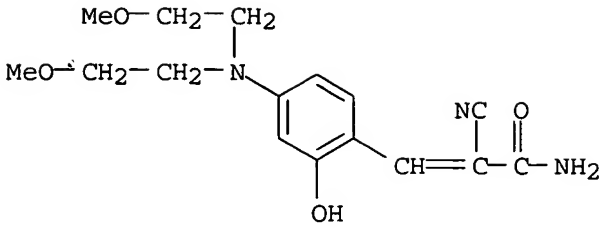
RN 50745-32-9 CAPLUS
CN Propanediamide, 2-[[4-(diethylamino)-2-hydroxyphenyl]methylene]- (9CI) (CA INDEX NAME)



RN 50745-33-0 CAPLUS
CN 2-Propenamide, 2-cyano-3-[4-(dimethylamino)-2-hydroxyphenyl]- (9CI) (CA INDEX NAME)



RN 50745-34-1 CAPLUS
CN 2-Propenamide, 3-[4-[bis(2-methoxyethyl)amino]-2-hydroxyphenyl]-2-cyano- (9CI) (CA INDEX NAME)



L4 ANSWER 63 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN
ACCESSION NUMBER: 1973:516365 CAPLUS
DOCUMENT NUMBER: 79:116365
TITLE: Water-soluble styryl dyes
INVENTOR(S): Gmaj, Jan
PATENT ASSIGNEE(S): Instytut Przemyslu Organicznego
SOURCE: Pol., 4 pp.
CODEN: POXXA7
DOCUMENT TYPE: Patent
LANGUAGE: Polish
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

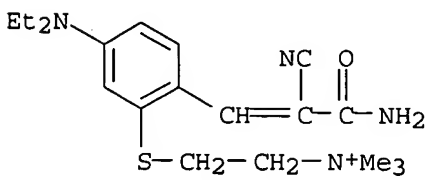
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PL 66159		19720731	PL	19690211 <--

AB Styryl dyes (I, R = CN, CONH2, R2 = Et, Me; Q = S, O; X = Cl, MeSO4, Cl . xZnCl2) were prepared and were used to dye polyacrylonitrile fiber light-, sublimation-, and washfast yellow shades. Thus, m-Et2NC6H4OCH2CH2NEt2 was treated with POCl3 in DMF to give 2-[2-(diethylamino)ethoxyl]-4-(diethylamino)benzaldehyde [42540-28-3] which was heated with CH2(CN)2, and treated with p-MeC6H4SO3Me to give styryl dye I(R = CN, R1 = Et, Q = O, X = Cl.xZnCl2, 2-substituted).

IT **50329-19-6P**
RL: IMF (Industrial manufacture); PREP (Preparation)
(preparation of)
RN 50329-19-6 CAPLUS
CN Ethanaminium, 2-[[2-(3-amino-2-cyano-3-oxo-1-propenyl)-5-(diethylamino)phenyl]thio]-N,N,N-trimethyl-, chloride, compd. with zinc chloride (ZnCl2) (9CI) (CA INDEX NAME)

CM 1

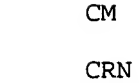
CRN 50582-78-0
CMF C19 H29 N4 O S . Cl



● Cl -

CM 2

CRN 7646-85-7
CMF Cl2 Zn

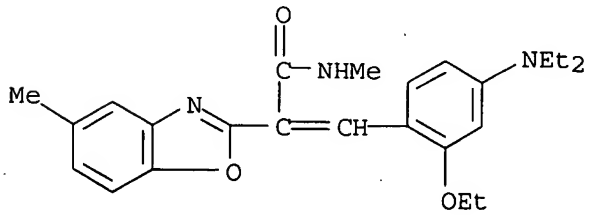


L4 ANSWER 64 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN
ACCESSION NUMBER: 1972:528075 CAPLUS
DOCUMENT NUMBER: 77:128075
TITLE: Oxazolylcoumarin dyes
INVENTOR(S): Harnisch, Horst
PATENT ASSIGNEE(S): Farbenfabriken Bayer A.-G.
SOURCE: Ger. Offen., 64 pp. Division of Ger. Offen 2,058,877.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2065076	A	19720622	DE 1970-2065076	19701130 <--
PRIORITY APPLN. INFO.:			DE 1970-2065076 A	19701130

AB Thirteen title compds. [I; R = Et or Me; R1 = Me, H, Cl, SO2NMe2, SO2Et, cyclohexyl, CMe3, or Ph; R2 = H, or (R1R2) = CH:CHCH:CH or o-C6H4O; R3 = H, Me, or SO3Na], dyeing polyester, polyamide, cellulose, and wool fibers fast brilliant greenish yellow shades, were prepared by reaction either of 2,4-HO(R2N)C6H3CHO (II) with benzoxazolylacetamides (III) (obtained from NCCH2CO2Et and R4R5NH via NCCH2CONR4R5 and subsequent cyclization with o-aminophenols) or of II with bis(benzoxazolyl)methanes (IV) and cyclization. Thus, NCCH2CO2Et and MeO(CH2)3NH2 were mixed with cooling and heated 30 min at 60.deg., 4,3-HO(H2N)C6H3Me was added, and the mixture heated 6 hr at 180.deg. under N to give the corresponding III, which without isolation was refluxed 20hr with II (R = Et) in Me2CHOH containing piperidine to give a dye (I; R = Et, R1 = Me, R2 = R3 = H) (V) [34564-13-1]. V was also obtained by reaction of 4,3-HO(H2N)C6H3Me and CH2(CO2Et)2 to give bis(5-methylbenzoxazolyl)methane [25798-47-4], reaction with II (R = Et) in EtOH containing piperidine to form 1-[2-hydroxy-4-(diethylamino)phenyl]-2,2-bis(5-methyl-2-benzoxazolyl)ethylene [36526-05-3], and cyclization with 96% H2SO4 at 50.deg..

IT **35773-52-5P**
RL: IMF (Industrial manufacture); PREP (Preparation)
(preparation of)
RN 35773-52-5 CAPLUS
CN 2-Benzoxazoleacetamide, α-[[4-(diethylamino)-2-ethoxyphenyl]methylene]-N,5-dimethyl- (9CI) (CA INDEX NAME)



L4 ANSWER 65 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN
ACCESSION NUMBER: 1972:528073 CAPLUS
DOCUMENT NUMBER: 77:128073
TITLE: Benzoxazolylcoumarin dyes and their 2-(2-benzoxazolyl)acetamide intermediates
INVENTOR(S): Harnisch, Horst
PATENT ASSIGNEE(S): Farbenfabriken Bayer A.-G.
SOURCE: Ger. Offen., 77 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2058877	A	19720615	DE 1970-2058877	19701130 <--
CH 717157	A4	19760630	CH 1971-7157	19710513 <--
CH 587833	A	19770513	CH 1973-16185	19710513 <--
CH 585250	A	19770228	CH 1973-16186	19710613 <--
BE 768722	A1	19711103	BE 1971-104800	19710618 <--
NL 7108436	A	19711222	NL 1971-8436	19710618 <--
GB 1329042	A	19730905	GB 1971-28704	19710618 <--
GB 1329043	A	19730905	GB 1972-38453	19710618 <--
JP 50023028	B4	19750805	JP 1971-43359	19710618 <--
US 3985763	A	19761012	US 1973-369124	19730612 <--
JP 50069380	A2	19750610	JP 1974-99075	19740830 <--
JP 51006266	B4	19760226		
JP 51000526	A2	19760106	JP 1974-99076	19740830 <--
JP 51042125	B4	19761113		

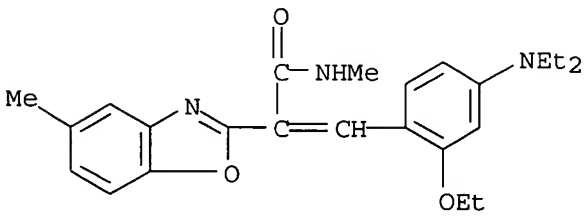
PRIORITY APPLN. INFO.: DE 1970-2030507 A 19700620
DE 1970-2058877 A 19701130
US 1971-154652 A1 19710618

AB Fourteen title dyes (I, R = e.g. H, Me, Cl, SO2NMe2, SO2Et, cyclohexyl; R1 = Me or Et), dyeing polyester, polyamide, cellulose triacetate, or wool fibers lightfast brilliant greenish shades, were prepared by reaction of (2-benzoxazolyl)acetamides (II) with 2,4-HO(R12N)C6H3CHO. Forty-seven II were prepared by reaction of o-aminophenols with NCCH2CONR2R3. For example, a mixture of NCCH2CO2Et and MeO(CH2)3NH2 was heated 30 min at 60.deg., 3,4-H2N(HO)C6H3Me added, and the mixture heated 6 hr at 180.deg. (bath temperature) to give the dye intermediate (II, R = Me, R2 = MeO(CH2)3, R3 = H) [35783-38-1] which was refluxed with 2,4-HO(Et2N)C6H3CHO, iso-PrOH, and piperidine for 20 hr to give a benzocoumarin dye (I, R = Me, R1 = Et) [34564-13-1].

IT **35773-52-5P**
RL: IMF (Industrial manufacture); PREP (Preparation)
(preparation of)

RN 35773-52-5 CAPLUS

CN 2-Benzoxazoleacetamide, α-[[4-(diethylamino)-2-ethoxyphenyl]methylene]-N,5-dimethyl- (9CI) (CA INDEX NAME)



L4 ANSWER 66 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN
ACCESSION NUMBER: 1972:128826 CAPLUS
DOCUMENT NUMBER: 76:128826
TITLE: Oxazolylacetic acid derivatives and oxazolylcoumarins for dyeing organic fibers
INVENTOR(S): Harnisch, Horst
PATENT ASSIGNEE(S): Farbenfabriken Bayer A.-G.
SOURCE: Ger. Offen., 80 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2030507	A	19720105	DE 1970-2030507	19700620 <--
DE 2030507	B2	19740919		
DE 2030507	C3	19750522		
CH 717157	A4	19760630	CH 1971-7157	19710513 <--

CH 587833	A	19770513	CH 1973-16185	19710513 <--
CH 585250	A	19770228	CH 1973-16186	19710613 <--
BE 768722	A1	19711103	BE 1971-104800	19710618 <--
NL 7108436	A	19711222	NL 1971-8436	19710618 <--
FR 2099247	A5	19720310	FR 1971-22352	19710618 <--
GB 1329042	A	19730905	GB 1971-28704	19710618 <--
GB 1329043	A	19730905	GB 1972-38453	19710618 <--
AT 310707	B	19731010	AT 1971-5278	19710618 <--
AT 310743	B	19731010	AT 1972-6152	19710618 <--
JP 50023028	B4	19750805	JP 1971-43359	19710618 <--
US 3985763	A	19761012	US 1973-369124	19730612 <--
JP 50069380	A2	19750610	JP 1974-99075	19740830 <--
JP 51006266	B4	19760226		
JP 51000526	A2	19760106	JP 1974-99076	19740830 <--
JP 51042125	B4	19761113		

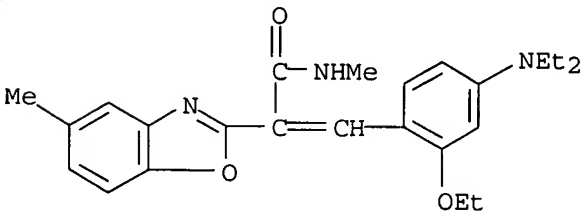
PRIORITY APPLN. INFO.: DE 1970-2030507 A 19700620
DE 1970-2058877 A 19701130
US 1971-154652 A1 19710618

AB Oxazoles [I, A represents benzene, naphthalene, or dibenzofuran ring; R = H, alkyl, cyclohexyl, aralkyl, aryl; R1 = H, alkyl, cyclohexyl, aralkyl, aryl, or (RR1N) = heterocyclic ring] were prepared by reaction of o-aminophenols with NCCH2CONRR1 and treated with 4-(dialkylamino)salicylaldehydes to give oxazolylcoumarins (II, R = Me, Et), fluorescent dyes for natural and synthetic fibers. For example, a mixture of o-H2NC6H4OH and NCCH2CONH2 was heated under N 30 min at 140-60.deg., 15 min at 150-60.deg., and 1 hr at 170.deg. to give 2-(2-benzoxazolyl)acetamide [34564-12-0]. Similarly, 46 other I were prepared A mixture of NCCH2CO2Et and MeO(CH2)3NH2 was heated 30 min at 60.deg., 3,4-H2N(HO)C6H3Me added, and the mixture heated 6 hr at 180.deg. to give N-(3-methoxypropyl)-5-methyl-2-benzoxazoleacetamide which (without isolation) was refluxed 20 hr with 4,2-Et2N(HO)C6H3CHO and iso-PrOH in the presence of piperidine to give 7-(diethylamino)-3-(5-methyl-2-benzoxazolyl)coumarin [34564-13-1], dyeing nylon-6 fabric a fast, brilliant greenish yellow shade. Similarly, 13 other II were prepared

IT **35773-52-5P**
RL: IMF (Industrial manufacture); PREP (Preparation)
(preparation of)

RN 35773-52-5 CAPLUS

CN 2-Benzoxazoleacetamide, α-[[4-(diethylamino)-2-ethoxyphenyl]methylene]-N,5-dimethyl- (9CI) (CA INDEX NAME)



L4 ANSWER 67 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN
ACCESSION NUMBER: 1972:87161 CAPLUS
DOCUMENT NUMBER: 76:87161
TITLE: Disperse methine dyes
INVENTOR(S): Kesler, Martin L.
PATENT ASSIGNEE(S): Martin-Marietta Corp.
SOURCE: Ger. Offen., 22 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2114574	A	19711014	DE 1971-2114574	19710325 <--

BE 764523	A1	19710816	BE 1971-101140		19710319 <--
NL 7103923	A	19710928	NL 1971-3923		19710324 <--
FR 2085111	A5	19711217	FR 1971-10470		19710324 <--

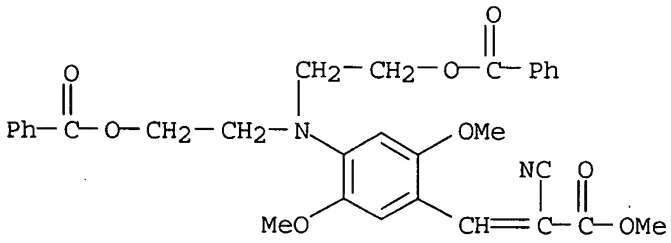
PRIORITY APPLN. INFO.: US 1970-22718 A 19700325

AB The title dyes (I, R = CN, CO2Et, or CO2Me, R1 = MeO or Me, R2 = MeO or H) were prepared by formylation of 2,5-R1R2C6H3N(CH2CH2OBz)2 with DMF and subsequent reaction with NCCH2R. I were used for dyeing poly(ethylene terephthalate) fibers bright greenish yellow shades. Thus, BzCl was added to 2,5-(MeO)2C6H3N(CH2OH)2 in pyridine, the mixture heated 30 min at 85-90.deg., extracted with H2O, residual H2O removed by azeotroxic distillation with PhMe, DMF and POCl3 were added, and the mixture was heated 30 min at 90-5.deg. to give 2,5,4-(MeO)2(HCO) C6H2N(CH2CH2OBz)2, which on refluxing with CH2(CN)2 4 hr in MeOH gave 4-(2,2-dicyanovinyl)-2,5-dimethoxy-N,N-bis(benzoyloxyethyl)aniline (I, R = CN, R1 = R2 = MeO) [34367-96-9]. Four other I were also prepared

IT **35473-19-9P 35473-20-2P**
RL: IMF (Industrial manufacture); PREP (Preparation)
(preparation of)

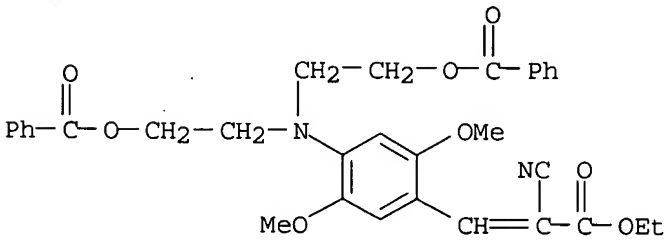
RN 35473-19-9 CAPLUS

CN 2-Propenoic acid, 3-[4-[bis[2-(benzoyloxy)ethyl]amino]-2,5-dimethoxyphenyl]-2-cyano-, methyl ester (9CI) (CA INDEX NAME)



RN 35473-20-2 CAPLUS

CN 2-Propenoic acid, 3-[4-[bis[2-(benzoyloxy)ethyl]amino]-2,5-dimethoxyphenyl]-2-cyano-, ethyl ester (9CI) (CA INDEX NAME)



L4 ANSWER 68 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN

ACCESSION NUMBER: 1971:552980 CAPLUS
DOCUMENT NUMBER: 75:152980
TITLE: Styryl dyes
INVENTOR(S): Enomoto, Shigeharu
PATENT ASSIGNEE(S): Sumitomo Chemical Co., Ltd.
SOURCE: Jpn. Tokkyo Koho, 6 pp.
CODEN: JAXXAD
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 46019710	B4	19710602	JP	19680117 <--

GI For diagram(s), see printed CA Issue.

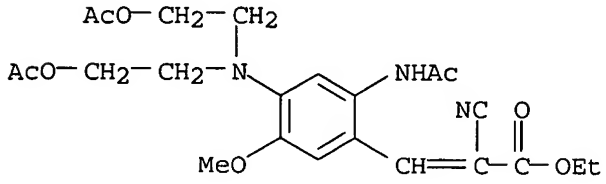
AB Yellow to greenish yellow styryl dyes (I, R = CN, CO2Me, CO2Et, R1 = Me, Ph, R2 = H, OMe) for polyester and cellulose acetate fibers were prepared by

condensation of RCH2CN and benzaldehydes in EtOH with an amine catalyst. 2-Acetamido-4-[bis(2-acetoxyethyl)amino]-β-(ethoxycarbonyl)-β-cyano-5-methoxystyrene and two other I were prepared

IT **34309-86-9P**
RL: IMF (Industrial manufacture); PREP (Preparation)
(preparation of)

RN 34309-86-9 CAPLUS

CN Cinnamic acid, 2-acetamido-4-[bis(2-hydroxyethyl)amino]-α-cyano-5-methoxy-, ethyl ester, diacetate (ester) (8CI) (CA INDEX NAME)



L4 ANSWER 69 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN

ACCESSION NUMBER: 1971:127560 CAPLUS

DOCUMENT NUMBER: 74:127560

TITLE: 3-Substituted-7-aminocoumarins, as optical brighteners or their intermediates

PATENT ASSIGNEE(S): Farbenfabriken Bayer A.-G.

SOURCE: Fr. Demande, 12 pp.
CODEN: FRXXBL

DOCUMENT TYPE: Patent

LANGUAGE: French

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE	
FR 2016308		19700508			<--
DE 1793262			DE		
GB 1230299			GB		
US 3681397		19720000	US		<--

PRIORITY APPLN. INFO.: DE 19680823

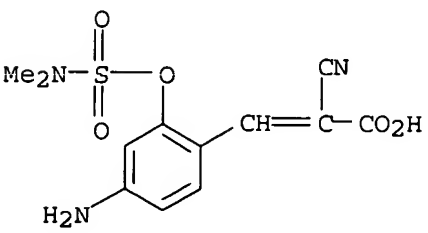
GI For diagram(s), see printed CA Issue.

AB Title compds. (I) are prepared by heating the corresponding 5,2,4-R1(RO)(H2N)C6H2CH:C(R2)CN (II, R = SO2NMe2 or CH2OMe) (cf. Fr. Demande 2,016,307) with aqueous mineral acids at 104-55° for 6-10 hr. Thus II (R1 = H, R2 = Ph, R = Me2NSO2) was heated in 62% H2SO4 at 130° for 7 hr to give I (R1 = H, R2 = Ph). Similarly 10 other I were prepared

IT **31804-49-6P**
RL: IMF (Industrial manufacture); PREP (Preparation)
(preparation of)

RN 31804-49-6 CAPLUS

CN Cinnamic acid, 4-amino-α-cyano-2-hydroxy-, dimethylsulfamate (ester) (8CI) (CA INDEX NAME)



L4 ANSWER 70 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN

ACCESSION NUMBER: 1971:113267 CAPLUS

DOCUMENT NUMBER: 74:113267
TITLE: Substituted β-phenylacrylonitrile derivatives as intermediates for optical brighteners
PATENT ASSIGNEE(S): Farbenfabriken Bayer A.-G.
SOURCE: Fr. Demande, 13 pp.
CODEN: FRXXBL
DOCUMENT TYPE: Patent
LANGUAGE: French
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE	
FR 2016307		19700508			<--
DE 1793261			DE		
GB 1272269			GB		
US 3890364		19750000	US		<--

PRIORITY APPLN. INFO.: DE 19680823

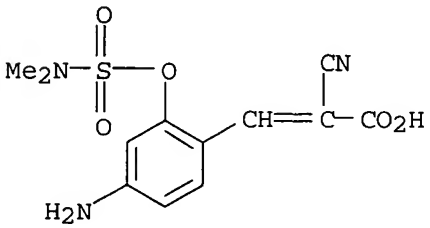
GI For diagram(s), see printed CA Issue.

AB 5,2,4-R1(RO)(O2N)C6H2Me are treated with Na2Sx in ROH or Me2SO at 50-120° 0.5-3 hr to give 5,2,4-R1-(RO)(H2N)C6H2CHO, which react with R2CH2CN at 20-120° to give the title products (I) which can be converted into the corresponding coumarins by heating with aqueous mineral acid at 80-200° (cf. Fr. Demande 2,016,308). Thus, an aqueous solution of Na2S, NaOH, and S was added dropwise to a boiling aqueous alc. solution of 2,4-MeO(O2N)C6H3Me, the mixture boiled for 0.5 hr, treated with PhCH2CN, and boiled for 1 hr to give I (R = Me, R1 = H, R2 = Ph). Similarly, 16 other I were prepared

IT **31804-49-6P**
RL: IMF (Industrial manufacture); PREP (Preparation)
(preparation of)

RN 31804-49-6 CAPLUS

CN Cinnamic acid, 4-amino-α-cyano-2-hydroxy-, dimethylsulfamate (ester) (8CI) (CA INDEX NAME)



L4 ANSWER 71 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN
ACCESSION NUMBER: 1971:65585 CAPLUS
DOCUMENT NUMBER: 74:65585
TITLE: 3-(4-Oxo-3,4-dihydro-2-quinazolinyl)-7-diethylaminocoumarin dyes
INVENTOR(S): Enomoto, Shigeharu; Sato, Katsunobu; Suzuki, Goichi
PATENT ASSIGNEE(S): Sumitomo Chemical Co., Ltd.
SOURCE: Ger. Offen., 53 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE	
DE 2005933	A	19701029	DE 1970-2005933	19700210	<--
DE 2005933	C3	19730726			
JP 48030333	B4	19730919	JP 1969-66793	19690822	<--
JP 48030450	B4	19730920	JP 1969-91217	19691113	<--
JP 48032409	B4	19731005	JP 1970-1868	19691227	<--

FR 2042045	A5	19710205	FR 1970-4704		19700210 <--
PRIORITY APPLN. INFO.:			JP 1969-30600	A	19690418
			JP 1969-66793	A	19690822
			JP 1969-79295	A	19691004
			JP 1969-91217	A	19691113
			JP 1970-1868	A	19691227

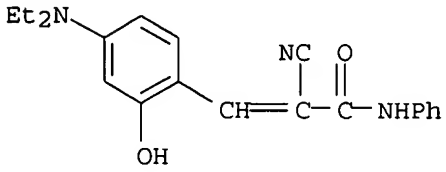
GI For diagram(s), see printed CA Issue.

AB The title dyes (I), where R = R1 = H and R2 = H, Me, MeO or R,R1 = CH:CHCH:CH and R2 = H, fluorescent yellow dyes for acetate, nylon, and polyester fibers, were prepared by reactio of coumarin-3-carboxylic acid derivs. with o-H2NC6H4-CONH2 (II), 4,2-Et2N(HO)C6H3CHO (III with 2-cyanomethyl-4(3H)-quinazolinones and subsequent cyclization, of 7-amino-3-carbamoylcoumarins with isatoic anhydride, or of III with acetanilides and subsequent cyclization with H2NCO2Et and P2O5. Thus heating Et 7-diethylaminocoumarin-3-carboxylate with II in Ph2 in the presence of B(OH)3 for 5-6 hr at 250-5° under N gave I (R = R1 = R2 = H). Similarly 3 other I were prepared

IT **30750-24-4P**, Cinnamanilide, α-cyano-4-(diethylamino)-2-hydroxy- **30750-26-6P**, Cinnamamide, α-cyano-4-(diethylamino)-2-hydroxy-N-1-naphthyl-
RL: IMF (Industrial manufacture); PREP (Preparation)
(preparation of)

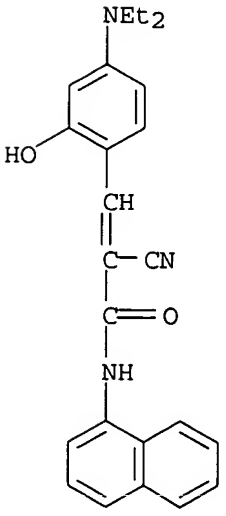
RN 30750-24-4 CAPLUS

CN Cinnamanilide, α-cyano-4-(diethylamino)-2-hydroxy- (8CI) (CA INDEX NAME)



RN 30750-26-6 CAPLUS

CN 2-Propenamide, 2-cyano-N-[4-(diethylamino)-2-hydroxyphenyl]-N-1-naphthalenyl- (9CI) (CA INDEX NAME)



L4 ANSWER 72 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN

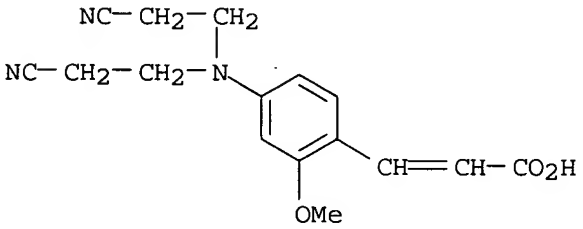
ACCESSION NUMBER:	1970:78584 CAPLUS
DOCUMENT NUMBER:	72:78584
TITLE:	Chemistry of bis(2-cyanoethyl) derivatives of some aromatic amines. V. Preparation of some new tertiary aminobenzaldehydes and a study of some of their reactions

AUTHOR(S): Jolly, V. S.; Ittyerah, P. I.
CORPORATE SOURCE: Chem. Lab., St. John's Coll., Agra, India
SOURCE: Journal of the Indian Chemical Society (**1969**), 46(11), 997-1002
CODEN: JICSAH; ISSN: 0019-4522
DOCUMENT TYPE: Journal
LANGUAGE: English

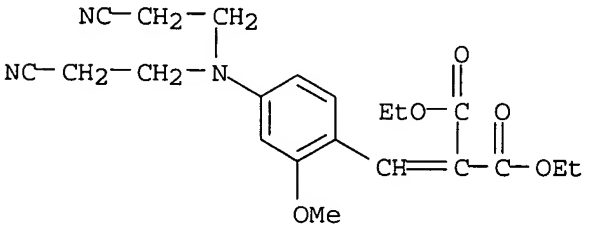
AB 4-[N,N-bis(2-cyanoethyl)amino]-2-ethoxy- and 2,6-(dimethylamino)benzaldehydes have been prepared for the first time. Some of the reactions of these aldehydes and also of 4-[N,N-bis-(2-cyanoethyl)amino]-2-methoxy- and 2-methylbenzaldehydes have been studied. p-[N-Methyl-N-(2'-cyanoethyl)amino]benzaldehyde which has so farbeen known through some of its derivs. has now been isolated in the pure form.

IT **28006-72-6P 28006-73-7P 28006-75-9P**
28006-79-3P 28006-81-7P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

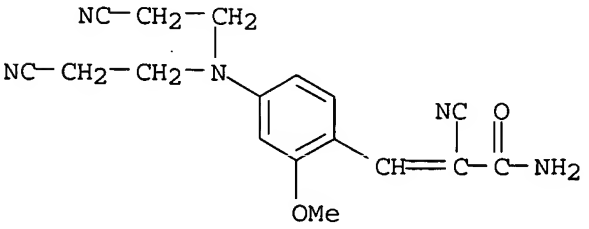
RN 28006-72-6 CAPLUS
CN Cinnamic acid, 4-[bis(2-cyanoethyl)amino]-2-methoxy- (8CI) (CA INDEX NAME)



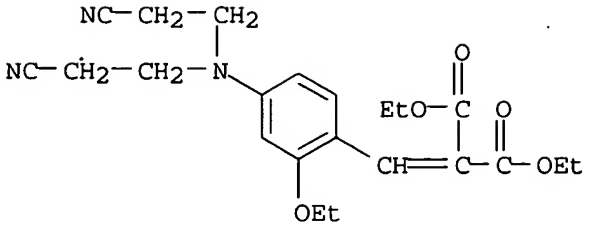
RN 28006-73-7 CAPLUS
CN Malonic acid, [4-[bis(2-cyanoethyl)amino]-2-methoxybenzylidene]-, diethyl ester (8CI) (CA INDEX NAME)



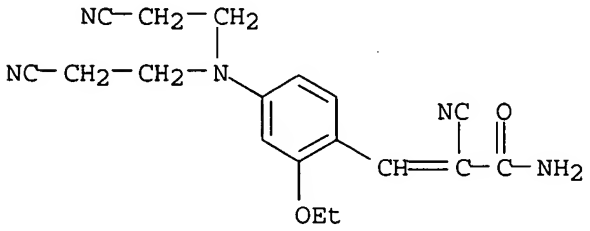
RN 28006-75-9 CAPLUS
CN Cinnamamide, 4-[bis(2-cyanoethyl)amino]-α-cyano-2-methoxy- (8CI) (CA INDEX NAME)



RN 28006-79-3 CAPLUS
CN Malonic acid, [4-[bis(2-cyanoethyl)amino]-2-ethoxybenzylidene]-, diethyl ester (8CI) (CA INDEX NAME)

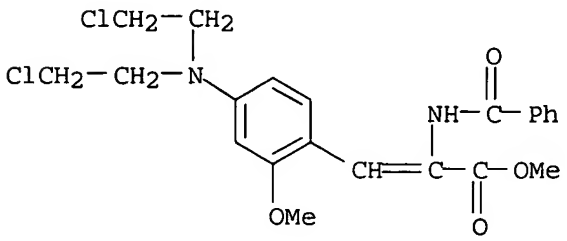


RN 28006-81-7 CAPLUS
CN Cinnamamide, 4-[bis(2-cyanoethyl)amino]-α-cyano-2-ethoxy- (8CI) (CA INDEX NAME)



L4 ANSWER 73 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN
ACCESSION NUMBER: 1968:50029 CAPLUS
DOCUMENT NUMBER: 68:50029
TITLE: Novel synthesis of o-methoxy-p-[Bis(2-chloroethyl)-amino]phenylalanine
AUTHOR(S): P'an, Pei-Ch'uan; Li, Tuan; Yao, Hsiao-Yu; Kao, I-Sheng
CORPORATE SOURCE: Inst. Mater. Med., Acad. Sinica, Shanghai, Peop. Rep. China
SOURCE: Yaoxue Xuebao (**1966**), 13(6), 432-7
CODEN: YHHPAL; ISSN: 0513-4870
DOCUMENT TYPE: Journal
LANGUAGE: Chinese
GI For diagram(s), see printed CA Issue.
AB Studies were made on the synthesis of title compound (I) which is active against carcinoma and some neoplastic diseases. 2,4-MeO[(RCH2CH2)2N]C6H3CH2C(NHCHO)(CO2Et)2 (II, R = Cl) (IIa) was obtained in 80% yield when II (R = OH) was treated with PCl5 in dry CH2Cl2. IIa thus obtained was pure enough to be hydrolyzed to I in satisfactory yield. A novel synthesis of I was also described: 3-MeOC6H4NH2 (III) (0.1 mole) was mixed with 0.5 mole ethylene oxide (IIIa) in 12.3 ml. dilute HOAc at 0° and allowed to stand 24 hrs. to give 99.8% 3-MeOC6H4N(CH2CH2OH)2 (IV), b0.6 180°, m. 49-50°. IV may also be prepared by refluxing 10.7 g. III with 68 ml. 30% ClCH2CH2OH and 13.5 g. CaCO3. IV (21 g.) dissolved in 46 ml. of Me2NCHO was cooled to 0° and 26 ml. of POCl3 was added dropwise at <40°, and stirred for 3 hrs. to give 25.2 g. 3,4-MeO(OCH)C6H3N(CH2CH2Cl)2 (V), m. 96-7° (EtOH). By condensing 150 g. V with 144 g. hippuric acid in the presence of 44 g. NaAc and 300 ml. Ac2O at 95-100° 121.5 g. VI, m. 192-2.5°, was obtained. VI was heated with 1000 ml. MeOH to 60°, cooled to 17°, 556 ml. 8.4% KOH added and stirred at room temperature to give 297 g. 3,4-MeO[CH:C(CO2Me)NHCOPh]C6H3N(CH2CH2Cl)2(VII) m. 136° (MeOH). VII with Zn dust in glacial HOAc <16° with stirring gave 88% 3,4-MeO[CH2CH(CO2R)NHCOPh]C6H3N(CH2CH2Cl)2 (VIIIa,R = Me) (VIII).HCl m. 98-9° (MeOH). VII was also prepared by reduction with H and Pd/C in HOAc for 6 hrs. with a yield of 90%. VIII (10 g.) was refluxed with 100 ml. HCl 2 hrs., to give VIIIa (R = H) (IX) which was redissolved in the next 4 hrs. to give 5.6 g. I, m. 177° (decomposition) (MeOH). IX, m. 174°, was obtained in 62% yield. IX formed VIII readily when heated with MeOH and the Et ester, m. 113-14°, of IX was formed by dissolving IX in EtOH, heating and standing.
IT **17126-77-1P**

RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
RN 17126-77-1 CAPLUS
CN Cinnamic acid, α-benzamido-4-[bis(2-chloroethyl)amino]-2-methoxy-, methyl ester (8CI) (CA INDEX NAME)



L4 ANSWER 74 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN
ACCESSION NUMBER: 1967:47320 CAPLUS
DOCUMENT NUMBER: 66:47320
TITLE: Reactive dyes
INVENTOR(S): Boresch, Carl; Raue, Roderich
PATENT ASSIGNEE(S): Farbenfabriken Bayer A.-G.
SOURCE: Ger., 7 pp.
CODEN: GWXXAW
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 1229212		19661124	DE	19610307 <--

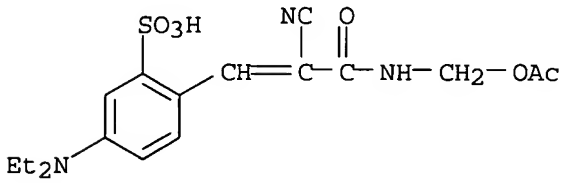
AB Azo, methine, and anthraquinone dyes containing a group of the formula -C(Y)N(R)CH(R1)O2CR2 (I) were prepared; in I, Y = O or NH, R and R1 = H or a substituent, and R2 is alkyl. The dyes, useful for dyeing cellulose fibers wetfast shades from an acid bath, were prepared by treatment of dyes containing a RNHC(Y) group with an aliphatic aldehyde and esterifying the resulting methylol compds. with an aliphatic carboxylic acid. Thus, a mixture of 5 parts 4-HO3SC6H4NH2 (II) → 1-phenyl-3-carbamoyl-5-pyrazolone (III), 1.5 parts paraformaldehyde (IV) and 15 parts AcOH (V) was heated at 80-5° for 40 min., 5 parts Ac2O added held at 80° for 10 min., cooled, and evaporated in vacuo to give a fast bright yellow dye for cotton. Similarly, the following dyes were prepared (starting dye, aldehyde, carboxylic acid, and shade on cotton given): II → 3-methyl-5-pyrazolone, IV, V, greenish yellow; 2,4-HO3S(Et2N)C6H3CH:C(CN)CONH2, IV, V, yellow; 1-amino-4-(4-carbamoylanilino)anthraquinone-2-sulfonic acid, IV, V, blue; 4-H2NCOC6H4NH2 (VI) → 1-(4-sulfophenyl)-3-methyl-5-pyrazolone, IV, EtCO2H (VII), reddish yellow; VI → 1,8,3,6,-HO(AcNH)C10H4(SO3H)2 (VIII), IV, VII, blush red; VI → 1,6,3,-HO(BzNH)C10H5SO3H (IX), IV, VII, yellowish red; II → 2,3-HOC10H6CONH2 (X)IV, VII, yellowish red; VI → VIII, Me-CHO, V, reddish violet; VI → 2,6-HOC10H6SO3H (XI), EtCHO, V, yellow-orange; VI → VIII, Cl3CHO.H2O, V, bluish-red; Cr complex of 2,3,5-HO(O2N)(HO3S)C6H2NH2 → III, IV, V, bluishred; VI → 1-(4-sulfophenyl)-3-methyl-5-pyrazolone (XII), IV, V, yellow; 4-MeNHCOC6H4NH2 → XII, IV, V, greenish yellow; [2,4-HO3S(H2N)C6H3CH2]2 → 2 moles III, IV, V, reddish yellow; VI → 1,6,3-HO(H2N)C10H5SO3H, IV, V, yellowish scarlet; VI → XI, IV, V, reddish orange; VI → VIII, IV, V, yellowish red; VI → IX, IV, V, red; methine dye from 1,3,3-trimethyl-2-methyleneindoline-5-sulfonic acid and 1-phenyl-3-carbamoyl-4-(dimethylaminomethylene)-5-pyrazolone, IV, V, yellowish orange, II → X, IV, V, red; 2-HO3SC6H4NH2 → X, IV, V, reddish orange; 2:1 Cr complex of 2,4-HO(HO3S)C6H3NH2 (XIII) → III, IV, V, bluish red; 2:1 Cr complex of XIII → X, IV, V, violet; 1-amino-4-(4-ureidoanilino)anthraquinone-2-sulfonic acid, IV, V, blue;

3-H2NCONHC6H4NH2 (XIV) → XII, IV, V, reddish yellow; 2:1 Cr complex of 2,3,5-HO(HO3S)(O2N)C6H2NH2 → III, IV, V, yellowish brown; 1-amino-4-(2-carbamoylanilino)anthraquinone-2-sulfonic acid, IV, V, reddish blue; 2-H2NCOC6H4NH2 → 1-(2-sulfophenyl)-3-methyl-5-pyrazolone (XV), IV, V, reddish yellow; XIV → XV, IV, V, reddish yellow; XIV → 1-(4,8-disulfonaphthyl)-3-methyl-5-pyrazolone, IV, V, greenish yellow.

IT **14662-66-9P**
RL: IMF (Industrial manufacture); PREP (Preparation)
(preparation of)

RN 14662-66-9 CAPLUS

CN Metanilic acid, 6-[2-cyano-2-[(hydroxymethyl)carbamoyl]vinyl]-N,N-diethyl-, acetate (ester) (8CI) (CA INDEX NAME)



L4 ANSWER 75 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN

ACCESSION NUMBER: 1966:103799 CAPLUS

DOCUMENT NUMBER: 64:103799

ORIGINAL REFERENCE NO.: 64:19469e-g

TITLE: Compounds with antiblastic activity. XXVIII. 2,5-Dimethoxy-4-[N,N-bis(β-chloroethyl)amino]benzaldehyde and its derivatives

AUTHOR(S): Artico, Marino; De Martino, Giovanni; Giuliano, Raffaele

CORPORATE SOURCE: Univ. Rome

SOURCE: Annali di Chimica (Rome, Italy) (**1966**), 56(174-81), 1-2
CODEN: ANCRAI; ISSN: 0003-4592

DOCUMENT TYPE: Journal

LANGUAGE: Italian

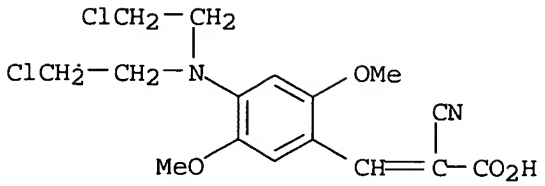
GI For diagram(s), see printed CA Issue.

AB cf. CA 64, 19435c. The title compound (I) and its condensation products with active methylene compds. were prepared for testing as tumor-inhibiting agents. Heating 0.4 mole 2,5-dimethoxyaniline and 0.7 mole ClC2H4OH to 130-40° 6 hrs. while slowly adding 0.8 mole 8% aqueous NaOH gave 83% N,N-bis(β-hydroxyethyl)-2,5-dimethoxyaniline (II), b0.2 145-8°. Adding 0.3 mole II in 150 ml. HCONMe2 to 1 mole POCl3 dissolved in 2 moles HCONMe2 and heating the mixture 3 hrs. at 90° gave 55% 1, m. 92-4° (EtOH), thiosemicarbazone decompose 203-4° (EtOH). I (0.01 mole) and 0.01 mole 2-methyl-3-carboxycyclopentanone in 20 ml. EtOH were treated during 5 min. at 60-5° with 0.03 mole KOH dissolved in 5 ml. H2O and 15 ml. EtOH, the mixture heated 5 min. and kept 45 min. to give 2-methyl-3-carboxy-5-[2,5-dimethoxy-4-[N, N-bis(β-chloroethyl)-amino]benzylidene]cyclopentanone, III (R = Me), m. 162-4° (MeOH). Similarly prepared was III (R = Et), m. 134-6°. I (0.01 mole) and 0.01 mole 4-O2NC6H4CH2CN in 15 ml. dioxane treated with cooling with 0.2 ml. Et2NH or piperidine and kept overnight at room temperature gave 90% IV (R = 4-NO2C6H4), m. 150-1° (CHCl3-petr. ether). Similarly prepared were IV (R = CO2Et), m. 122-4° (CHCl3-Et2O) and IV (R = CO2H), m. 210-11° (CHCl3).

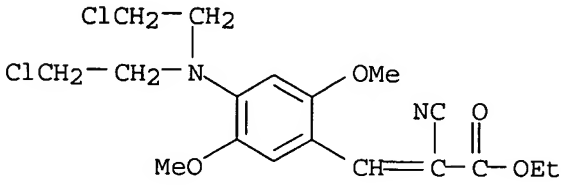
IT **5551-05-3**, Cinnamic acid, 4-[bis(2-chloroethyl)amino]-α-cyano-2,5-dimethoxy- **5611-92-7**, Cinnamic acid, 4-[bis(2-chloroethyl)amino]-α-cyano-2,5-dimethoxy-, ethyl ester
(preparation of)

RN 5551-05-3 CAPLUS

CN Cinnamic acid, 4-[bis(2-chloroethyl)amino]-α-cyano-2,5-dimethoxy- (7CI, 8CI) (CA INDEX NAME)



RN 5611-92-7 CAPLUS
CN 2-Propenoic acid, 3-[4-[bis(2-chloroethyl)amino]-2,5-dimethoxyphenyl]-2-cyano-, ethyl ester (9CI) (CA INDEX NAME)



L4 ANSWER 76 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN
ACCESSION NUMBER: 1963:486261 CAPLUS
DOCUMENT NUMBER: 59:86261
ORIGINAL REFERENCE NO.: 59:5096b-h,5097a-e
TITLE: Wilting agents and antibiotics. XXVIII Synthesis of 2,4 dimethoxy 6 hydroxyphenanthrene and constitution of orchinol.
AUTHOR(S): Hardegger, E.; Biland, H. R.; Corrodi, H.
CORPORATE SOURCE: Eidg. Tech. Hochschule, Zuerich, Switz.
SOURCE: Helv. Chim. Acta (**1963**), 46, 1354-60
DOCUMENT TYPE: Journal
LANGUAGE: German
GI For diagram(s), see printed CA Issue.
AB (All m.ps. are corrected). 3,5 (MeO)2C6H3CH2CN [prepared from tech. α-resorcylic acid via 3,5-(MeO)2C6H3CO2H] (160 g.) refluxed 16 h. with 1.6 l. 20% aqueous KOH and the solution cooled, filtered, extracted with a little Et2O, and acidified with concentrated HCl gave 160 g. 3,5-(MeO)2C6H3CH2CO2H (I), m. 100-1°. I (30 g.) and 30 g. 2,4(O2N)2C6H3CHO dissolved in 300 mL. Ac2O, the solution treated with 21.5 mL. Et3N (the temperature rose to 40-50°), kept 16 h., concentrated in vacuo (H2O pump) at 50-60° to 50-75 mL., treated with 75 mL. H2O at 90° with vigorous shaking, the precipitate filtered off, washed with H2O, dried in vacuo, boiled with 100 mL. C6H6, filtered off while hot, and dried gave 37 g. 2,4 (O2N)2C6H3CH: C[C6H3(OMe)2-3,5]CO2R (II) (R = H), m. 205-6° (C6H6). II (R = H) (3.75 g.) suspended in 200 mL. Et2O treated with Et2OCH2N2 until all solid dissolved and N evolution ceased, the solution evaporated, the residue chromatographed on Al2O3 (activity II), and the product eluted with C6H6 gave 3.9 g. II (R = Me), needles, m. 95-6° (Et2O-MeOH); sometimes II (R = Me) was obtained as rhombohedrons, m. 118°; seeding an Et2O solution of the low melting ester with crystals of the higher melting ester gave quant. higher melting ester. II (R = Me) (3.88 g.) in 200 mL. MeOH hydrogenated over 500 mg. 10% Pd-C (after 1 h. and 22 h. 1600 mL. H and 1710 mL. H, resp., was absorbed), the solution filtered, evaporated in vacuo, and the residual oil (3.3 g.) treated with MeOH gave α-(3,5 dimethoxyphenyl)-β-(2,4 diaminophenyl)propionic acid δ-lactam, m. 185° (CHCl3-MeOH); Ac derivative m. 256-8° (CHCl3-MeOH). II (R = H) (10 g.) dissolved in 150 mL. hot AcOH, the solution treated with 18.2 g. SnCl2.2H2O in 30 mL. AcOH at 20° with stirring, saturated with HCl at 0°, stirred 24 h., concentrated in vacuo at 40° to 30 mL., dissolved in 200 mL. Et2O, the solution washed with 7 50-mL. portions H2O until the wash H2O was colorless, extracted with 3 50-mL. portions 2N NaOH, the combined exts. acidified with concentrated HCl, the product isolated with CH2Cl2, dissolved in 50 mL. EtOH, and the solution treated with HCl, the product, α-(3,5 dimethoxyphenyl) 2 amino 4 nitrocinnamic acid HCl salt (III.HCl), m. 70° (decomposition), filtered off, treated with 150 mL. 1:1 EtOH-H2O,

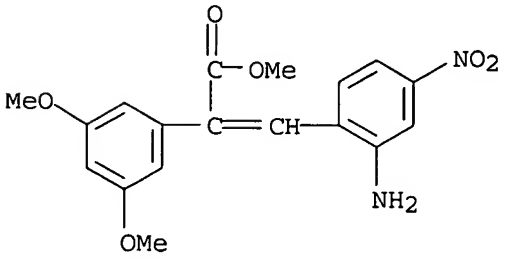
the mixture boiled until a clear solution formed, and the solution concentrated to 75 mL. and cooled gave 2.7 g. III, m. 205° (CHCl3-MeOH). III treated with CH_2N_2 in Et_2O, the product chromatographed on Al_2O_3 (activity II), and the column eluted with CH_2Cl_2 and Et_2O gave Me ester of III, m. 172° (MeOH-CHCl8). III (2.4 g.) dissolved in 36 mL. concentrated H_2SO_4 at -10°, the solution poured on 130 g. ice, treated during 15 min. with 1.45 mL. 5N $NaNO_2$ at 0° with stirring, stirred 1.5 h., treated with 100 mL. H_2O, stirred 1.5 h., treated with a small amount of urea (after 0.5 h. HNO_2 was no longer detectable with KI-starch paper), filtered through Celite, the filter cake washed with H_2O until no reaction with β-naphthol was obtained, the combined filtrates concentrated by boiling 45 min. at 100°, the precipitate filtered off, esterified with CH_2N_2, the product chromatographed on Al_2O_3 (activity II), and the column eluted with C_6H_6 gave 1.13 g. 2,4 dimethoxy 6 nitro 10 phenanthrenecarboxylic acid (IV) Me ester (V), m. 198° (C_6H_6). V (20 mg.) in 10 mL. MeOH boiled 2 h. with 2 mL. N KOH, diluted with 20 mL. H_2O, and acidified with a few drops concentrated HCl gave IV, m. 280-1° (decomposition) (CH_2Cl_2-MeOH). V (1.04 g.) in 125 mL. THF hydrogenated over 1 g. prereduced 10% Pd-C (after 10 min. 190 mL. H absorbed, after 3 h. 207 mL. H, and finally 228 mL. H after 1 min. after addition of 500 mg. prereduced 10% Pd-C) gave 6-NH_2 analog (VI) of V, m. 147-8° (MeOH). VI (622 mg.) dissolved in 20 mL. concentrated H_2SO_4 at -10°, the solution poured on 100 g. ice with shaking, the resulting suspension treated during 16 min. with 2.1 rel. N $NaNO_2$ at 0°, the mixture stirred 2 h. at 0°, diluted with 100 mL. H_2O, stirred 2 h., treated with urea, stirred 0.5 h. (excess HNO_2 was now destroyed), heated 0.5 h. at 100°, cooled, the precipitate (650 mg.) filtered off, boiled 3 h. with 20 mL. MeOH and 5 mL. H_2O containing 1 g. KOH, the solution evaporated, the residue dissolved in 20 mL. H_2O, the solution acidified with concentrated HCl, the precipitate filtered off, decarboxylated by boiling 2.5 h. in 10 mL. quinoline with 100 mg. Cu chromite, the mixture added to 100 mL. 2N HCl, filtered, the filter cake and filtrate extracted with Et_2O, the combined exts. evaporated, the residual oil (235 mg.) chromatographed on Al_2O_3 (activity II), the column cluted with C_6H_6-Et_2O, and the product (63 mg.) crystallized from C_6H_6-hexane gave 8 mg. 2,4-dimethoxy-6hydroxyphenanthrene (VII), m. 135°. VI (311 mg.) diazotized and the solution of diazonium salt boiled down as above, the precipitate (350 mg.) filtered off, treated in 100 mL. MeOH with excess Et_2O-CH_2N_2, after cessation of N evolution the solution evaporated, the residual oil (378 mg.) chromatographed on Al_2O_3 (activity II), and the product eluted with C_6H_6 gave 54 mg. Me 2,4,6-trimethoxy-10-phenanthrenecarboxylate, m. 130-1°, which was saponified and decarboxylated as above and then chromatographed on Al_2O_3 (activity II) and eluted with C_6H_6 to give 17 mg. 2,4,6trimethoxyphenanthrene (VIII), m. 109-10° (hexane). Dehydroorchinol (m. 168-70°) was different from synthetic VIII (m. 136°). Although the m.ps. of dehydroorchinol Me ether (m. 113-14°) and synthetic VIII (m. 109-10°) differed only slightly, the mixed m.p. was depressed by 25-30°. From this, it followed that orchinol (Villa) was 2,4-dimethoxy-7-hydroxy-9,10-dihydrophenanthrene. As a supplement to the synthesis of VII was mentioned another route (see below) which, although not carried to completion, should also lead to VII. 2,3,5, 6-Br(MeO)2(O_2N) C_6HCHO (29 g.) dissolved in 900 mL. hot Ac_2O, the solution treated with 30.5 g. p-$HOC_6H_4CH_2CO_2H$ and 14 mL. Et_3M at 20°, kept 6 h. at 95-100° with periodic shaking, concentrated in vacuo to 50 mL., heated to 90° with 50 mL. H_2O, evaporated in vacuo, the residual solid dried 6 h. in vacuo, heated to boiling with 150 mL. C_6H_6, and the solution filtered and evaporated gave 20.2 g. 2,3,5,6-Br(MeO)2(O_2N)C_6HCH:C(C_6H_4OR-4)CO_2R' (IX) (R = R' = H) (X), m. 263-6° (slight decomposition above 230°) (dioxane). X (3 g.) suspended in 200 mL. MeOH treated with Et_2O-CH_2N_2 (all solid dissolved) gave IX (R = H, R' = Me), m. 210-11° (MeOH) X (4.3 g.) in a little H_2O treated portionwise during 1 h. with 18 mL. 4N KOH and 3.8 g. Me_2SO_4 at 100° with stirring in such a way that the mixture always remained alkaline, the whole stirred 0.5 h. at 100°, diluted with H_2O, filtered, and the filtrate acidified with. 2N HCl gave 3.5 g. IX (R = Me, R' = H), m. 224° (EtOHCCl4). X (3.47 g.) in 100 mL. EtOH refluxed 21 h. with 3 g. K_2CO_3 and 2.5 mL. $PhCH_2Cl$, the solution filtered, evaporated, the residue dissolved in 300 mL. N Na_2CO_3, the solution washed with Et_2O, brought to pH 1-2 with concentrated HCl, and the product isolated with $CHCl_3$ gave 1.8 g. IX (R =

PhCH2, R' = H), m. 2357° (CHCl3-MeOH), which was treated in 1:1 MeOH-Me2CO with Et2O-CH2N2 to give IX (R = PhCH2, R' = Me), m. 187° (Et2O-petr. ether).

IT **93880-29-6**, Acrylic acid, 3-(2-amino-4-nitrophenyl)-2-(3,5-dimethoxyphenyl)-, methyl ester **97980-08-0**, Acrylic acid, 3-(2-amino-4-nitrophenyl)-2-(3,5-dimethoxyphenyl)-, hydrochloride
(preparation of)

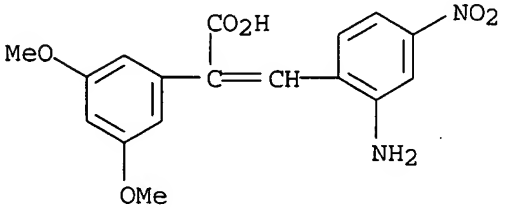
RN 93880-29-6 CAPLUS

CN Acrylic acid, 3-(2-amino-4-nitrophenyl)-2-(3,5-dimethoxyphenyl)-, methyl ester (7CI) (CA INDEX NAME)



RN 97980-08-0 CAPLUS

CN Acrylic acid, 3-(2-amino-4-nitrophenyl)-2-(3,5-dimethoxyphenyl)-, hydrochloride (7CI) (CA INDEX NAME)



● HCl

L4 ANSWER 77 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN

ACCESSION NUMBER: 1963:469602 CAPLUS

DOCUMENT NUMBER: 59:69602

ORIGINAL REFERENCE NO.: 59:12961a-f

TITLE: Azo and anthraquinone dyes

INVENTOR(S): Raue, Carl Boresch; Raue, Roderich

PATENT ASSIGNEE(S): Farbenfabriken Bayer A.-G.

SOURCE: 24 pp.

DOCUMENT TYPE: Patent

LANGUAGE: Unavailable

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE	
BE 614660		19620905	BE		<--
GB 971920			GB		
US 3261827		1966	US		<--
PRIORITY APPLN. INFO.:			DE	19610307	

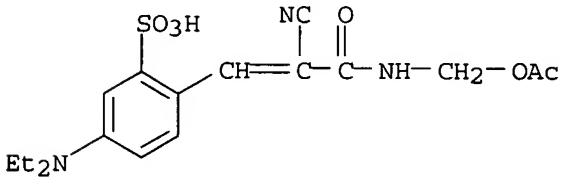
AB Dyes containing carboxamide groups are treated with an aldehyde and an organic acid to give compds. containing C(:O)N(R)CH(R')OC(:O)R'' groups (R = H or a substituent, R' = H or alkyl; R'' = Me or Et) which dye cotton and cellulose textiles. Thus, a mixture of the dye (p-H2NC6H4SO3H → 1-phenyl-5-pyrazolone-3-carboxamide) 5, paraformaldehyde (I) 1.5, and HOAc 15 is heated at 80-5° for 40 min., Ac2O 5 parts added, and the mixture heated at >80° for 10 min., cooled, filtered, and the filtrate evaporated in vacuo at 40° to give a dye. The prepared dye (30

parts) is dissolved in 1000 parts H2O containing HOAc, and cotton is impregnated with the solution, treated (foulard) to 70%, fixed at 140° for 15 min., and rinsed and soaped to give a bright yellow dyeing with good wet-and lightfastness. Other dyes are similarly prepared (compound treated with I and HOAc, shade on cotton given): p-H2NC6H4SO8H → 3-methyl-5-pyrazolone, greenish yellow; reaction product of p-H2NC6H4CONH2 (II) with 1-amino-4-bromoanthra-quinone-2-sulfonic acid, blue; p-H2NC6H4SO3H → 1-phenylpyrazolone-3-carboxamide (III), reddish yellow; p-H2NC6H4CONHMe → 1-(p-sulfophenyl)-3-methylpyrazolone (IV), greenish yellow; [4,2-H2N(HO3S)C6H3CH2]2 two stacked rightwards arrow II, reddish yellow; II → 1,6,3-HO(H2N)(HO3S)C10H5, yellowish scarlet; II → 2,6-HO(HO3S)C10H6, reddish orange; II → 1,8,3,6-HO(AcNH)-(HO3S)2C10H4 (V), wine red; II → 1,6,3-HO(BzNH)(HO3S)-C10H5, reddish yellow; II → IV, yellow; reaction product of 1,3,3-trimethyl-2-methyleneindolene-5-sulfonic acid with 1-phenyl-4-(dimethylaminomethylene)pyrazolone-3-carboxamide, yellowish orange; p-H2NC6H4SO3H → 2,3-HO(H2NCOC10H6, bright red; o-H2NC6H4SO3H → 2,3-HO(H2NCO)C10H6, yellowish orange; 1:2 Cr complex of [3,4-HO(H2N)C6H3SO14H (VI) → III], slightly bluish red; 1:2 Cr complex of [VI → 2,3-HO(H2NCO)C10H6], violet; 1-amino-4-(m-ureidoanilino)anthraquinone-2-sulfonic acid, blue; 3-H2NC6H4NHCONH2 → IV, reddish yellow; 1:2 Cr complex of [2,3,5-HO(HO3S)(O2N)C6H2NH2 → III, yellowish brown; 1-amino-4-(o-carbamoylanilino)anthraquinone-2-sulfonic acid, reddish blue; 2-H2NC6H4CONH2 → 1-(2-sulfophenyl)-3-phenylpyrazolone, reddish yellow; 1-(o-sulfophenyl)-3-methyl-4-(m-ureidophenylazo)-5-pyrazolone, reddish yellow; Cr complex of [2,4,6-HO(HO3S)(O2N)C6H2NH2 → III], bluish red; 3-H2NC6H4NHCONHH2 → 1-(4,8-disulfonaphthyl)-3-methylpyrazolone, greenish yellow. Also prepared are the following dyes (reactant, aldehyde, acid, color on cotton given): II → V, AcH, HOAc, reddish violet; II → 2,6-HO(HO3S)C10H6, EtCHO, HOAc, orange yellow; II → V, Cl3CCHO.H2O, HOAc, bluish red; II → IV, I, EtCO2H, reddish yellow; also prepared is 4-(m-sulfophenylazo)-1-(acetoxymethyl)-3-methyl-5-pyrazolone, greenish yellow on cotton.

IT **14662-66-9**, Metanilic acid, 6-[2-cyano-2-[(hydroxymethyl)carbamoyl]vinyl]-N,N-diethyl-, acetate
(preparation of)

RN 14662-66-9 CAPLUS

CN Metanilic acid, 6-[2-cyano-2-[(hydroxymethyl)carbamoyl]vinyl]-N,N-diethyl-, acetate (ester) (8CI) (CA INDEX NAME)



L4 ANSWER 78 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN

ACCESSION NUMBER: 1963:428381 CAPLUS

DOCUMENT NUMBER: 59:28381

ORIGINAL REFERENCE NO.: 59:5094g-h,5095a-h,5096a-b

TITLE: Wilting agents and antibiotics. XXVII. Induced defensive substances in the Orchidaceae. 2

AUTHOR(S): Hardegger, E.; Schellenbaum, M.; Corrodi, H.

CORPORATE SOURCE: Eidg. Tech. Hochschule, Zuerich, Switz.

SOURCE: Helvetica Chimica Acta (**1963**), 46, 1171-80
CODEN: HCACAV; ISSN: 0018-019X

DOCUMENT TYPE: Journal

LANGUAGE: German

GI For diagram(s), see printed CA Issue.

AB Biol. investigations have shown that under the influence of certain morbific agents, defensive substances are produced in the corms of Orchidaceae; e.g., the mycorrhizal fungus Rhizoctonia repens activates defense mech anisms in the corms of Orchis militaris, which clearly result

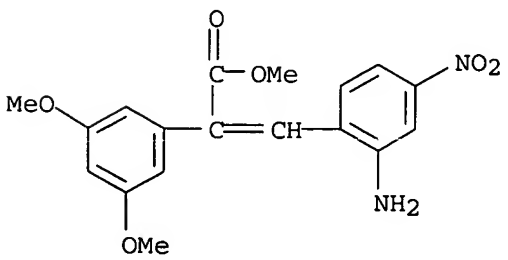
in the formation of orchinol (I), C16H16O3, as the sole defensive substance, along with biol. inactive p-HOC6H4CH2OH (II); both I and II are not found in healthy plants. However, Loroglossum hircinum produces no I, but other defensive substances against R. repens. From infected corms of L. hircinum was isolated a biol. inactive compound, C16H16O3, designated lorroglossol (III), isomeric with and closely related to I. (All m.ps. are corrected). The Et2Oeluate (loc. cit.) chromatographed again on Al2O3 (activity II) gave II, m. 120° (MeOH-H2O), mol. weight (camphor) 136. pHOC6H4CO2Me (10 g.) in 150 mL. Et2O added dropwise to 6 g. LiAlH4 in 100 mL. Et2O at 20° with stirring, the whole refluxed 3 h., decomposed with EtOAc and H2O under ice cooling, acidified with AcOH, and the product isolated with Et2O gave 2 g. II, m. 122° (H2O). I (20 γ) in 20 μl. MeOH applied to Whatman Number 1 paper, the solution allowed to travel with 1:1 MeOH-H2O, the paper dried, sprayed with 0.1% alc. N,2,6 trichloro-p-benzoquinone imine, followed by saturated aqueous borax, and dried gave a grayish green spot corresponding to I with Rf 0.56; I had Rf 0.79 with 1:1 EtOH-H2O. EtOH-Et2O-exts. of infected corm fragments of L. hircinum were prepared and worked up in a manner similar to the isolation of I from the corms of O. militaris to give III, m. 98° (C6H6cyclohexane, then MeOH). III (50 mg.), 0.1 mL. Me2SO4, and 140 mg. K2CO3 in 10 mL. Me2CO refluxed 22 h., cooled, filtered, the filtrate evaporated, the residue (52 mg.) chromatographed on Al2O3 (activity I), and the column eluted with CH2Cl2 gave 27 mg. Me ether of III, b0.1 200°. I (20 mg.) and 93 mg. 3,5(O2N)2C6H3COCl in 1 mL. absolute pyridine kept 30 min. at 20°, boiled 2 min., cooled, diluted with 20 mL. Et2O, filtered, the filtrate washed with dilute HCl, saturated aqueous KHCO3, and saturated salt solution, dried, and evaporated gave 30 mg. I 3,5 dinitrobenzoate, m. 198° (CH2Cl-Et2O). A solution of 500 mg. I, 1.9 g. p-MeC6H4SO2Cl (IIIa), and 5 mL. pyridine was prepared at 0°, kept 24 h. at 20°, treated with 1 mL. H2O, kept 1 h., taken up in CHCl3, and the solution washed (dilute HCl, saturated aqueous KHCO3, and H2O) and evaporated to give 774 mg. I tosylate (IV), oil which crystallized, m. 101-3° (MeOH-H2O). IV (50 mg.) and 25 mg. NaI in Me2CO or in Ac2O refluxed 5 h. gave (from each experiment) quant. unchanged IV. IV (100 mg.) and 100 mg. LiAlH4 in 5 mL. dioxane refluxed 2 h., treated with EtOAc and H2O to destroy excess LiAlH4, acidified with AcOH, and the product isolated with Et2O (the extract was washed in the usual manner) gave 59 mg. I after crystallization from C6H6-cyclohexane. Saponification of IV with dilute aqueous NaOH also gave I. I (300 mg.) in 6 mL. Et2O and 21 mL. 2% Et2O-CH2N2 kept 12 h. at 20°, the solution filtered, evaporated, the residue chromatographed on Al2O3 (activity II), and the column eluted with C6H6 gave 51 mg. Me ether (V) of I, m. 86-7° (cyclohexane); continued elution with 1:1 C6H6-Et2O gave 213 mg. unchanged I. I (340 mg.) stirred to a paste with a little H2O, the paste treated during 1 h. with alternate portions of 2.3 mL. 4N KOH (total) and 0.37 mL. Me2SO4 (total) at 100° in such a way that the mixture always remained alkaline, kept 30 min. at 100°, cooled, filtered, the filtrate extracted with C6H6, the extract washed, evaporated, and the residue purified as above gave 298 mg. V, m. 86-7°. To 128 mg. I in 2 mL. AcOH was added dropwise 80 mg. Br in 1 mL. AcOH and the solution poured into H2O to give di-Br derivative of I, m. 154° (CCl4). To 200 mg. I in 4 mL. CHCl3 and 10 mL. CCl4 was added dropwise during 30 min. 9 mL. 0.18 M CCl4-Br at 0°, the solution stirred 30 min. (no more free Cl was present) evaporated in vacuo, the residue (265 mg.) adsorbed on silica gel, the chromatogram developed with C6H6CHCl3, the column extruded, and the visible zones sectioned and eluted with CHCl3 to give 153 mg. di-Cl derivative of I, m. 133-40° (unsharp) (C6H6-cyclohexane, then sublimation in vacuo), and 63 mg. tri-Cl derivative of I, m. 198-9° (C6H6-cyclohexane, then sublimation in vacuo); the former compound migrated slower than the latter compound IV (750 mg.) in 60 mL. EtOH hydrogenated at atmospheric pressure over 4 g. fresh prereduced Raney Ni W-2, the hydrogenation continued (2 addns. of 2 g. fresh catalyst were made) (after 3 days 128 mL. H was absorbed), the solution filtered, evaporated, the partially crystalline residue dissolved in C6H6, the solution washed with H2O, evaporated, the residue (286 mg.) chromatographed on Al2O3 (activity I), and the column eluted with C6H6 gave 1st 58 mg. oil and then 228 mg. deoxyorchinol (VI), C16H16O2, m. 58-9° (pentane). VI (170 mg.) and 510 mg. pyridine-HCl heated 6 h. at 210-20°, the mixture partitioned between Et2O-2N HCl, and the Et2O-layer washed (H2O and 2N NaOH) and evaporated gave 11 mg. neutral oily

fraction; the NaOH-soluble product (135 mg.) chromatographed on silica gel and the product eluted with Et2O gave 117 mg. deoxydidemethylorchinol (VII), m. 145° (C6H6). o- (VIII) and pC6H4(OH)2 (IX) and VII (5 mg. each) in absolute Et2O and in absolute C6H6 were boiled 5 min. with 500 mg. Ag2O and kept overnight. VIII and IX gave instantaneous red and yellow colors, resp., with Et2O (even at 20°). VII did not give these color reactions. VII (100 mg.) and 0.2 mL. Ac2O in 1 mL. pyridine kept 12 h. at 20° and poured into ice H2O gave 117 mg. VII diacetate, m. 92-3° (C6H6-petr. ether). VII (75 mg.) and 665 mg. IIIa in 2 mL. pyri- dine kept 12 h. at 20° and worked up as was iV gave 173 mg. VII ditosylate, m. 163° (C6H6-Et2O). I (500 mg.) and 75 mg. 10% Pd-C heated 5 min. at 180-200° (34 mL. H obtained), the product chromatographed on Al2O3 (activity II), and the column eluted with 1:1 C6H6-Et2O gave 266 mg. dehydroorchinol (X), C16H14O3, m. 168-70° (C6H6). X (100 mg.) methylated with 0.11 mL. Me2SO4 and 0.7 mL. 4N KOH as above, the product chromatographed on Al2O3 (activity II), and the column eluted with 1:1 C6H6-petr. ether gave 94 mg. X Me ether (XI), m. 113-14°. V (540 mg.) and 80 mg. 10% Pd-C heated 5 h. at 210-80° (31 mL. H obtained), the product chromatographed on Al2O3 (activity II), eluted with C6H6-petr. ether, and recrystd. from C6H6-petr. ether gave 340 mg. XI, m. 111-13°; unchanged V remained in the mother liquor. VII (250 mg.) and 40 mg. 10% Pd-C heated 5 h. at 250-300° (6 mL. H and an undetd, amount H2O obtained), the petr. ethersol. fraction of the dehydrogenation product chromatographed on Al2O3 (activity I), and the column eluted with petr. ether gave 54 mg. phenanthrene, m. 94-5° (EtOH) [trinitrobenzene complex m. 158° (EtOH)]; the petr. ether insol. fraction recrystd. from C6H6-petr. ether gave 2 phenanthrol, m. 163-4° [acetate m. 139-40° (C6H6-petr. ether)]. VI (300 mg.) and 45 mg. 10% Pd-C heated 1 h. at 260-80° (21 mL. H obtained), the C6H6-soluble fraction of the dehydrogenation product chromatographed on Al2O3 (activity I), and the product eluted with 1:1 C6H6-petr. ether and repeatedly recrystd. from cyclohexane gave 146 mg. deoxydehydroorchinol (XII), C16H14O2, m. 75-6°, identical (mixed m.p. and UV and IR spectra) with 2,4 dimethoxyphenanthrene. XII (107 mg.) and 320 mg. pyridine-HCl heated 6 h. at 210-20°, the product (CHCl3-soluble, H2O-insol.) extracted with 2N NaOH, the extract acidified, the resulting oil (77 mg.) acetylated with Ac2O in pyridine, and this product chromatographed in silica gel and eluted with Et2O gave 52 mg. di O acetyldeoxydehydrodidemethylorchinol (XIII), m. 128-30°. These results indicated that I was either 2,4 dimethoxy 6 or 7 hydroxy 9,10 dihydrophenanthrene (XIV). The UV spectrum (EtOH) of I and the IR spectra (KBr) of I and XII were recorded.

IT **93880-29-6**, Acrylic acid, 3-(2-amino-4-nitrophenyl)-2-(3,5-dimethoxyphenyl)-, methyl ester **97980-08-0**, Acrylic acid, 3-(2-amino-4-nitrophenyl)-2-(3,5-dimethoxyphenyl)-, hydrochloride **412323-20-7**, Acrylic acid, 3-(2-amino-4-nitrophenyl)-2-(3,5-dimethoxyphenyl)-
(preparation of)

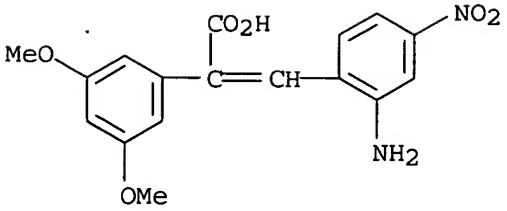
RN 93880-29-6 CAPLUS

CN Acrylic acid, 3-(2-amino-4-nitrophenyl)-2-(3,5-dimethoxyphenyl)-, methyl ester (7CI) (CA INDEX NAME)



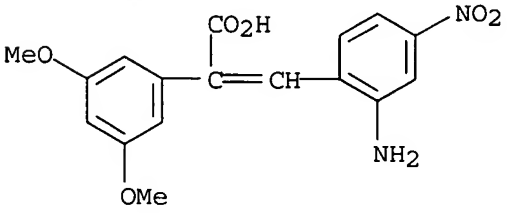
RN 97980-08-0 CAPLUS

CN Acrylic acid, 3-(2-amino-4-nitrophenyl)-2-(3,5-dimethoxyphenyl)-, hydrochloride (7CI) (CA INDEX NAME)



● HCl

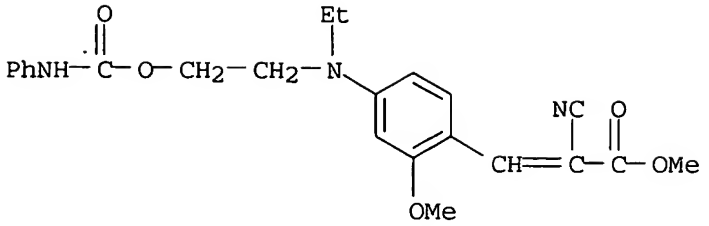
RN 412323-20-7 CAPLUS
CN Benzeneacetic acid, α-[(2-amino-4-nitrophenyl)methylene]-3,5-dimethoxy- (9CI) (CA INDEX NAME)



L4 ANSWER 79 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN
ACCESSION NUMBER: 1962:469785 CAPLUS
DOCUMENT NUMBER: 57:69785
ORIGINAL REFERENCE NO.: 57:13936d-f
TITLE: Water-insoluble styryl dyes
INVENTOR(S): Merian, Ernest; Nicolaus, Bruno J. R.; Senn, Otto
PATENT ASSIGNEE(S): Sandoz Ltd.
SOURCE: 7 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CH 358534		19620115	CH	19570712 <--

GI For diagram(s), see printed CA Issue.
AB Styryl derivs. of formula I, where R = Me or Et, and X = CO2Me, CN, or p-MeC6H6SO2, prcpd. by condensing benzaldehyde derivs. with acetonitrile derivs., are valuable greenish yellow dyes for coloring lacquers, oils, resins, and polymers from organic solns. and for dyeing polyamide, acetate silk, polyacrylonitrile, and terephthalate fibers from dispersions, in greenish yellow shades of good fastness properties. Thus, 4-[N-ethyl-N-(2-phenylcarbamoyloxyethyl)amino]-2-methylbenzaldehyde 32.6 and MeO2CCH2CN 10 are refluxed with piperidine 1 and MeOH 30 parts and the dark-yellow mass cooled to 0° to give I, R = Et, X = CO2Et, m. 122°. Similarly prepared was I, R = Et, X = p-MeC6H4SO2, m. 145°.
IT **95442-01-6**, Cinnamic acid, α-cyano-4-[ethyl(2-hydroxyethyl)amino]-2-methoxy-, methyl ester carbanilate
(preparation of)
RN 95442-01-6 CAPLUS
CN Cinnamic acid, α-cyano-4-[ethyl(2-hydroxyethyl)amino]-2-methoxy-, methyl ester carbanilate (7CI) (CA INDEX NAME)



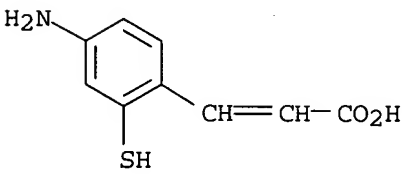
L4 ANSWER 80 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN
ACCESSION NUMBER: 1959:45191 CAPLUS
DOCUMENT NUMBER: 53:45191
ORIGINAL REFERENCE NO.: 53:8131i,8132a-i,8133a
TITLE: 2-Nitro-4-aminobenzaldehyde and thiocoumarin derivatives. I
AUTHOR(S): Ricci, Adolfo
CORPORATE SOURCE: Univ. Perugia, Italy
SOURCE: Annali di Chimica (Rome, Italy) (**1958**), 48, 985-96
CODEN: ANCRAI; ISSN: 0003-4592
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
GI For diagram(s), see printed CA Issue.
AB cf. C.A. 51, 16454i. Preparation of derivs. of 2,4-O2N(H2N)C6H8CHO (I) is described; these are to be tested for bacteriostatic properties. Cyclization of 2,4-HS(H2N)C6H3CH:CHCO2H (II) gives 7-aminothiacoumarin (III) from which a series of fluorescent thiacoumarins are prepared These are being tested for photo-dynamic activity and action against paramecium. 2,4-O2N(AcNH)C6H3Me (10 g.) in 80 cc. Ac2O and 100 cc. AcOH cooled to 0°, treated slowly with 11 cc. H2SO4 below 10° then with 14 g. CrO3 in 80 cc. Ac2O at 15-20°, kept 1 hr., and drowned in ice H2O ppts. 50% 2,4-O2N(AcNH)C6H3CH(OAC)2, m. 146-7°, hydrolyzed by HCl in aqueous EtOH to 85% I, m. 140-1°. A high-melting, insol. polymer of I is precipitated at the same time and during recrystn. of I. I (5 g.) and 2 g. MeNO2 in EtOH at -5° is treated with 3.5 g. KOH in 6.5 cc. H2O and 65 cc. EtOH, kept 15 min. at -5°, then filtered to give 2,4-O2N(H2N)C6H3CH(OH)CH2NO2, m. 138-45° (unstable), boiled 5 min. with 2 g. NaOAc and 20 cc. Ac2O then drowned in H2O to give 2,4-O2N(AcNH)C6H3CH:CHNO2, m. 187-8° (decomposition). I (10 g.) added to 8 g. barbituric acid in 80 cc. H2O gives a black precipitate, insol. in most solvents, extracted with dioxane to leave yellow 5-(2-nitro-4-aminobenzylidene)barbituric acid, not m. 360°. I forms a thiosemicarbazone (IV), m. 255-6°. IV (2 g.) is refluxed several hrs. with 0.9 g. succinic anhydride in xylene, cooled, filtered, the precipitate dissolved in hot Na2CO3, and cooled to precipitate the Na salt of 2-nitro-4-(succinylamino)-benzaldehyde thiosemicarbazone; the free acid, m. 228° (decomposition). IV (2 g.) refluxed 12 hrs. in EtOH with 0.8 g. ClCH2CO2H and 1.6 g. NaHCO3, concentrated, diluted with H2O, and acidified ppts. 2,4-O2N(HO2CCH2NH)C6H3CH:NNHCSNH2, m. 279° (decomposition). I (5 g.) in 20 cc. HCO2H is treated with 8 ml. concentrated HCl, diazotized at 0° with 2.1 g. NaNO2 in H2O, the solution poured into 3.6 g. CuSCN and 17.5 g. KSCN in a min. of H2O, heated to complete the reaction, diluted with 10 vols. H2O, and filtered to give 2,4-O2N(NCS)C6H3CHO, m. 108°. Reduction of 5 g. I in hot aqueous EtOH by 60 g. FeSO4 and 30 ml. NH4OH at 60-70° gives 35-40% 2,4-(H2N)2C6H3CHO, m. 152° (thiosemicarbazone, m. 225-6°). I (10 g.) and 10 g. CH2(CO2H)2 in 25 cc. EtOH is refluxed 4 hrs. with 1 ml. pyridine, filtered, and the filtrate concentrated to give a 2nd crop of 2,4-O2N(H2N)C6H3CH:CHCO2H, m. 255-6° (decomposition); Ac derivative, m. 280-1° (decomposition). This (2 g.) in 6 cc. HCl is reduced at 60-70° by 3.4 g. Sn to 7-aminocarbostyril (V), m 290-1°. Reduction of 10 g. 2,4-O2N(AcNH)C6H3CH:CHCO2H by FeSO4-NH4OH gives 2,4-H2N(AcNH)C6H3CH:CHCO2H (VI), m. 228° (decomposition), hydrolyzed by acid to V. VI (10 g.) in 50 cc. HCO2H (d. 1.20) is treated with 11.5 cc. HCl (HCl salt precipitated), diazotized, and poured into a solution of 6 g. CuSCN and 27 g. KSCN to give 2,4-NCS(AcNH)C6H3CH:CHCO2H, m. 207-8°. This (5 g.) is treated with

1.7 g. $NaHCO_3$ in a little H_2O, then with 5 g. Na_2S, heated 1 hr. at 50-60°, then cooled, and acidified to precipitate II, m. 210-12°. II (5 g.) and 10 g. NaOAc is heated 1 hr. in 25 cc. Ac_2O, diluted with H_2O, kept several hrs., filtered, the precipitate washed with warm aqueous Na_2CO_3 and H_2O, dissolved in boiling dilute HCl, the solution concentrated, and cooled to precipitate III.-HCl, filtered off, dissolved in H_2O, and treated with $NaHCO_3$ to precipitate III, m. 176-7°, volatile in steam. III (2 g.) dissolved in hot H_2O containing 3 cc. concentrated HCl, cooled, diazotized, poured into 1.2 g. CuCl in concentrated HCl, diluted and heated, then made alkaline, and steam distilled gives 7-chlorothiacoumarin, m. 136.5°. Similarly are prepared 7-iodo-(m. 141-2°) and 7-cyanothiacoumarin (m. 231-2°). III (2 g.) in 4 cc. HCO_2H is treated with 1 cc. concentrated H_2SO_4, diazotized, poured into 1.6 g. CuBr in concentrated HBr, diluted, heated, and filtered to give 7-bromothiacoumarin, m. 105-6°. 7-Thiocyanothiacoumarin, m. 154-5°, is prepared similarly. III (2 g.) is dissolved in 2 cc. concentrated H_2SO_4 in 100 cc. hot H_2O, cooled, diazotized, heated slowly to 70-80° and finally refluxed then cooled to precipitate 7-hydroxythiacoumarin, m. 231-2°. This is methylated by MeI in 2N KOH to 7-methoxythiacoumarin, m. 108° (30% unchanged compound recovered). III (2 g.) in 10 cc. AcOH is treated with 2.3 g. powdered KSCN then dropwise with 0.6 ml. Br in 10 ml. AcOH, kept 30 min., then poured into 200 ml. H_2O. The precipitate (a mixture of 6(?)-thiocyano-7-aminothiacoumarin and VII) is boiled with 2N HCl, concentrated, and made alkaline with Na_2CO_3 to precipitate VII, m. 293-4°.

IT **99357-80-9**, Cinnamic acid, 4-amino-2-mercapto- **100060-72-8**, Cinnamic acid, 4-acetamido-2-amino- **117000-64-3**, Cinnamic acid, 4-acetamido-2-thiocyanato-
(preparation of)

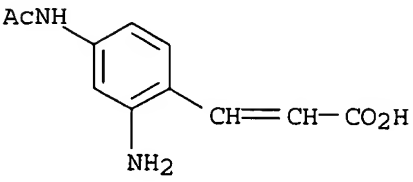
RN 99357-80-9 CAPLUS

CN Cinnamic acid, 4-amino-2-mercapto- (6CI) (CA INDEX NAME)



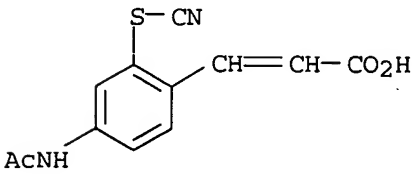
RN 100060-72-8 CAPLUS

CN Cinnamic acid, 4-acetamido-2-amino- (6CI) (CA INDEX NAME)



RN 117000-64-3 CAPLUS

CN Cinnamic acid, 4-acetamido-2-thiocyanato- (6CI) (CA INDEX NAME)



L4 ANSWER 81 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN

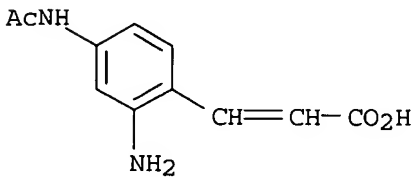
ACCESSION NUMBER: 1959:41180 CAPLUS

DOCUMENT NUMBER: 53:41180

ORIGINAL REFERENCE NO.: 53:7423e-f
TITLE: Antibacterial potency of styrene derivatives I
AUTHOR(S): Ricci, Adolfo; Angeletti, Pietro U.
CORPORATE SOURCE: Univ. Perugia, Italy
SOURCE: Bollettino Chimico Farmaceutico (**1958**), 97, 662-7
CODEN: BCFAAI; ISSN: 0006-6648
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB 2-Nitro-4-acetamido-β-nitro-styrene (I) was bacteristatic against Staphylococcus aureus at concns. of 5 γ/ml., which activity increased by increased concentration to 15 γ/ml. The organisms were completely inhibited at higher concentration of I after 18 hrs. of incubation. The substance was less effective against Escherichia coli. Three cinnamic acid derivs. had insignificant activity. Intraperitoneal injections of 20 mg./kg. I in mice were well tolerated.

IT **100060-72-8**, Cinnamic acid, 4-acetamido-2-amino-
(effect on bacteria)
RN 100060-72-8 CAPLUS
CN Cinnamic acid, 4-acetamido-2-amino- (6CI) (CA INDEX NAME)

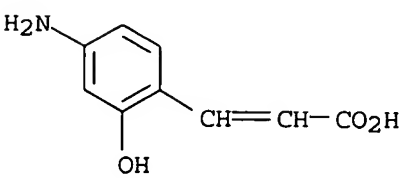


L4 ANSWER 82 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN
ACCESSION NUMBER: 1958:92937 CAPLUS
DOCUMENT NUMBER: 52:92937
ORIGINAL REFERENCE NO.: 52:16373f-g
TITLE: p-Aminocoumaric acid
INVENTOR(S): Libermann, D.
PATENT ASSIGNEE(S): Chimie et atomistique
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 1057860		19540311	FR	<--

AB p-Aminocoumaric acid (I) is useful as a bacteriostatic and tuberculostatic agent in veterinary medicine. Thus, 2.5 g. Na is dissolved in 15 ml. EtOH, and 1.6 g. 7-aminocoumarin is added. After 10 min. refluxing, the solution is allowed to stand several hrs. at room temperature, evaporated at room temperature, and the residue taken up in H2O and acidified by AcOH. The precipitate is dissolved in dilute NH3 and repptd. with AcOH to give I, m. 181° (decomposition).

IT **99357-85-4**, Cinnamic acid, 4-amino-2-hydroxy-
(preparation of)
RN 99357-85-4 CAPLUS
CN Cinnamic acid, 4-amino-2-hydroxy- (6CI) (CA INDEX NAME)

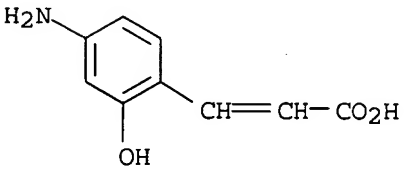


L4 ANSWER 83 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN
ACCESSION NUMBER: 1958:92936 CAPLUS
DOCUMENT NUMBER: 52:92936
ORIGINAL REFERENCE NO.: 52:16373d-f
TITLE: 3,5-Dioxopyrazolidine derivatives
INVENTOR(S): Wiedemann, O.
PATENT ASSIGNEE(S): J. R. Geigy A.-G.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
IL 9097		19560913	IL	<--

AB The title compds. are prepared by treating a reactive derivative of a monosubstituted malonic acid with a metal organic compound of an azobenzene at room temperature or by heating under reflux. EtBr (30.5 g.) in 60 ml. absolute ether was slowly added to 6.8 g. Mg in 20 ml. ether, the mixture boiled under reflux 30 min., treated dropwise with 25.5 g. (PhN:)2 in 200 ml. absolute ether while cooling in ice H2O, repeatedly shaken, boiled for 30 min. more under reflux, and cooled to -10° to give a pale brown powder. Butylmalonyl chloride (I) (27.6 g.) in 200 ml. absolute ether was added slowly at 0-5° with shaking, to this mixture, the whole boiled 2 hrs. under reflux and left standing for a day, to give a mixture containing a tough brown resin in the ether solution Acidifying and working up gave after recrystn. from alc. 1,2-diphenyl-3,5-dioxo-4-butylpyrazolidine, m. 106°, also obtained by treating I with N,N'-di-lithiohydrazobenzene.

IT **99357-85-4**, Cinnamic acid, 4-amino-2-hydroxy-
(preparation of)
RN 99357-85-4 CAPLUS
CN Cinnamic acid, 4-amino-2-hydroxy- (6CI) (CA INDEX NAME)



L4 ANSWER 84 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN
ACCESSION NUMBER: 1957:74459 CAPLUS
DOCUMENT NUMBER: 51:74459
ORIGINAL REFERENCE NO.: 51:13409g-i,13410a-b
TITLE: Methine dyes for synthetic fibers
INVENTOR(S): Kartinos, Nicholas J.; Normington, James B.; Williams, Wm. W.
PATENT ASSIGNEE(S): General Aniline & Film Corp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2789125		19570416	US	<--

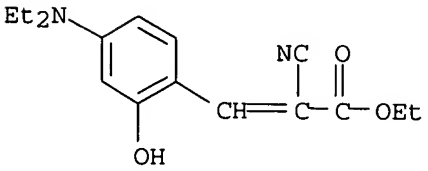
AB Products, having high tinctorial strength, excellent light-, chlorine-, and wash-fastness, good sublimation and fluorescent properties, and adaptability as fluorescent pigments and brightening agents, particularly for synthetic fibers, such as acetate rayons, are obtained by condensing a 2-substituted 4-[dialkyl- or bis(alkylcarboxyalkyl)amino]benzaldehyde with an alkyl cyanoacetate or cyanoethyl cyanoacetate in the presence of a basic or acid condensing agent. The dyes have the formula 2,4-R'(R2N)C6H3CH:C(CN)CO2CH2CH2CN, where R is a lower alkyl group, and R' is a halogen, hydroxy, or lower alkoxy group. 2-Ethoxy-4-

diethylaminobenzaldehyde (I), m. 45.8°, was obtained in 38% yield by combining 96.5 g. of N,N-diethyl-m-phenetidine and 73 g. of dimethylformamide, cooling to 10°, adding 92 ml. of POCl3 dropwise during 45 min., warming on a steam bath for 4 hrs., cooling, drowning in ice water, and adding 300 ml. of 40% NaOH solution until the pH was 3-5. By mixing 11.05 g. of I, 6.8 g. of Et cyanoacetate (II), 30 ml. of iso-PrOH (III), and 5 drops of piperidine (IV), mildly refluxing for 1 hr., collecting and drying the bright-orange solid gave Et α-cyano-4-(diethylamino)-2-ethoxycinnamate in 57% yield, m. 74-5°, and fluorescing strongly under ultraviolet light. The following derivs. of α-cyanocinnamate were also prepared: Et 4-(diethylamino)-2-hydroxy, m. 147-9°, from 2-hydroxy-4-diethylaminobenzaldehyde, m. 62°, and II; cyanoethyl 4-(diethylamino)-2-ethoxy, b0.7-0.8 150-4°, from I and cyanoethyl cyanoacetate; Et 4-(diethylamino)-2-methoxy from 2-methoxy-4-diethylaminobenzaldehyde and II; Et 4-(diethylamino)-2-chloro, m. 83.5°, from 2-chloro-4-diethylaminobenzaldehyde, b0.6 132-5°, and II; cyanomethyl 2-chloro-4-diethylamino, m. 98-100°; cyanoethyl 2-methyl-4-[bis(ethylcarboxyethyl)-amino], m. 122-4°; cyanoethyl 4-[bis(ethylcarboxyethyl)-amino], m. 104-8°; and Et 2-chloro-4-[bis(ethylcarboxyethyl)-amino], m. 64-5°. The essentially H2O-insol. dyes are applied directly to fabric as aqueous suspensions or dispersions.

IT **101586-75-8**, Cinnamic acid, α-cyano-4-diethylamino-2-hydroxy-, ethyl ester **101602-91-9**, Cinnamic acid, α-cyano-4-diethylamino-2-methoxy-, ethyl ester
(preparation of)

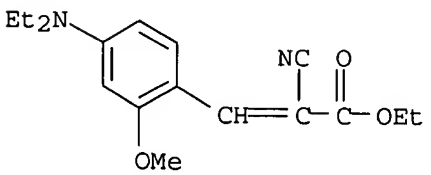
RN 101586-75-8 CAPLUS

CN Cinnamic acid, α-cyano-4-diethylamino-2-hydroxy-, ethyl ester (6CI) (CA INDEX NAME)



RN 101602-91-9 CAPLUS

CN 2-Propenoic acid, 2-cyano-3-[4-(diethylamino)-2-methoxyphenyl]-, ethyl ester (9CI) (CA INDEX NAME)



L4 ANSWER 85 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN

ACCESSION NUMBER: 1957:66507 CAPLUS

DOCUMENT NUMBER: 51:66507

ORIGINAL REFERENCE NO.: 51:12035a-i

TITLE: Reactions of amino acids and peptides with aromatic aldehydes. I

AUTHOR(S): Havinga, E.; Spitzer, E. L. T. M.

CORPORATE SOURCE: Univ. Leiden, Neth.

SOURCE: Recueil des Travaux Chimiques des Pays-Bas et de la Belgique (**1957**), 76, 173-9
CODEN: RTCPB4; ISSN: 0370-7539

DOCUMENT TYPE: Journal

LANGUAGE: English

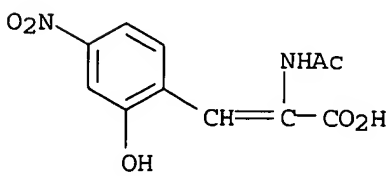
AB Formation of unsatd. azlactones by the Erlenmeyer-Plochl reaction with Ac2O as an acetylating medium and NaOAc as catalyst according to Dakin

(C.A. 23, 4205), and with EtOH as solvent in the absence of a catalyst by the method of Bergmann, et al. (C.A. 46, 8637e), has been investigated. Glycine (1.5 g.) heated in 3 ml. AcOH and 2 ml. Ac2O, the clear solution treated with 1.64 g. anhydrous NaOAc, 2.2 g. BzH, and 7 ml. Ac2O, heated 2 hrs. on a water bath at 95°, cooled, diluted with H2O, and the precipitate recrystd. from C6H6 gave α-acetamidocinnamic acid azlactone, m. 152-3°, which, heated in 0.5N NaOH with C, filtered, the filtrate acidified, and the product crystallized from H2O gave PhCH:C(NHAc)CO2H, m. 195-6°. Similarly, were prepared the following RCH:C(NHAc)CO2H (R and m.p. given): p-O2NC6H4, 227-9°; p-ClC6H4, 223-4°; 2,4-HO(O2N)C6H3, 218-20° (from BuOH-petr. ether); and the corresponding azlactones, m. 182-4°, 143-5° (fluorescent in ultraviolet light), and 298-310° (from AcOH). Glycine (1.5 g.) and 3.92 g. 2,4-(O2N)2C6H3CHO treated as above, the tarry product taken up in 0.5N NaOH, the solution heated, filtered, the filtrate acidified, and the precipitate crystallized from BuOH gave 2,4-(O2N)C6H3CH:C(NHAc)CO2H, m. 205-7°. NEt3 as an alternative to NaOAc did not affect the yields. Ascending paper chromatography with 21:39.5:39.5 pyridine-BuOH-H2O as eluant was used to follow the course of the reactions, the acetamidocinnamic acids giving dark spots (cf. Rydon and Smith, C.A. 46, 11290b), also detected under ultraviolet light by fluorescence or as dark spots. No "Dakin" condensation occurred with glycine derivs. in which the CO2H group had been esterified (cf. Doherty, et al., C.A. 38, 641), though acetylalanylglycine (I) gave a crystalline product. I (1.1 g.) added to 0.9 g. p-O2NC6H4CHO and 0.9 g. anhydrous NaOAc in 10 ml. hot Ac2O and 2 ml. AcOH, the cooled mixture filtered, the crystalline product (1.44 g.) taken up in H2O, filtered, and the residue twice extracted with EtOH and crystallized from dioxane gave a crystalline condensation product, C14H13N3O5, m. 210°, orange fluorescence in ultraviolet light. The above series of aldehydes, with the exception of 2,4-(O2N)2C6H3CHO, reacted readily with H2NCH2CO2Et (II) at room temperature in EtOH. The course of the reaction was followed by ascending paper chromatography with 40:10:50 BuOH-AcOH-H2O, in which the Schiff base of the condensation product hydrolyzes to phenylserine, detected by ninhydrin as well as by o-tolidine (cf. Reindel and Hoppe, C.A. 49, 4459d). II (2.06 g., freshly prepared) and 6.04 g. p-O2NC6H4CHO in 25 ml. absolute alc. heated 2 hrs. at 75°, the cooled mixture filtered, and the product crystallized from absolute EtOH gave 1.3 g. N-p-nitrobenzylidene-β-p-nitrophenylserine Et ester, m. 149-50°, decomposed by addition of HCl to a solution in alc. to β-p-nitrophenylserine Et ester HCl salt, m. 182°. The mother liquor treated with EtOH and HCl, the solution concentrated in vacuo, extracted with H2O, filtered, and the product crystallized from EtOH-EtOAc-Et2O gave the threo-isomer, m. 156-8° (cf. Holland, et al., C.A. 48, 10,680b). Similarly was obtained β-p-(chlorophenyl)serine Et ester HCl salt, m. 183° (from BuOH and EtOH). H2NCH2CONH2 (III) (350 mg.) and 1.4 g. p-O2NC6H4CHO dissolved in 25 ml. absolute alc. at 75°, the solution kept 3 days at room temperature and 12 hrs. at -8°, filtered, and the residue recrystd. from dioxane gave 370 mg. Schiff base of III; the mother liquor yielded 750 mg. 2nd crop, crystallized from HCONMe2 and dioxane to give N-p-nitrobenzylidene-β-p-nitrophenylserinamide, m. 183-5°. The ester group is therefore not essential for the condensation but since glycine esters substituted at the NH2 group failed to react with p-O2NC6H4CHO, a free NH2 group is essential for condensations under these conditions.

IT **99845-20-2**, Cinnamic acid, α-acetamido-2-hydroxy-4-nitro- (preparation of)

RN 99845-20-2 CAPLUS

CN Cinnamic acid, α-acetamido-2-hydroxy-4-nitro- (6CI) (CA INDEX NAME)



L4 ANSWER 86 OF 86 CAPLUS COPYRIGHT 2005 ACS on STN
ACCESSION NUMBER: 1955:12261 CAPLUS
DOCUMENT NUMBER: 49:12261
ORIGINAL REFERENCE NO.: 49:2505g-h
TITLE: Cinnamic acid derivatives
PATENT ASSIGNEE(S): Cilag Ltd.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CH 287557		19531016	CH	<--

AB Substituted cinnamic acid derivs. are produced by the interaction of diazonium salts with CH2:CHCO2H. Thus to 25.2 g. 4,2-O2N(MeO)C6H3NH2 in 350 ml. water and 42 ml. concentrated HCl diazotized with 10.8 g. NaNO2 and cooled to -5° is added 10.8 g. CH2:CHCO2H, 7.5 g. CuCl2, and 70 g. NaOAc, the mixture is stirred overnight, let stand for a day, the precipitate extracted with aqueous NaHCO3, the extract acidified, and purified by C yields 11-14 g. 4,2-O2N(MeO)C6H3CH:CHCO2H, m. 257-8°, reduced with Raney Ni and H in EtOH 6 hrs. at 20° to 8.5 g. 4-H2N analog, m. 160° (decomposition).

IT **195046-20-9**, Cinnamic acid, 4-amino-2-methoxy-
(preparation of)

RN 195046-20-9 CAPLUS

CN 2-Propenoic acid, 3-(4-amino-2-methoxyphenyl)- (9CI) (CA INDEX NAME)